

SNAP-8 MATERIALS REPORT FOR JANUARY - JUNE 1966

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LEWIS RESEARCH CENTER

CONTRACT NAS 5-417

FACILITY FORM 602	N67 18985	
	(ACCESSION NUMBER)	(THRU)
	10 126 153	1
	(PAGES)	(CODE)
	NASA-CR-72156	22
	(NASA CR OR TMX OR AD NUMBER)	(CATEGORY)



VON KARMAN CENTER
AEROJET-GENERAL CORPORATION
AZUSA, CALIFORNIA

NOTICE

This report was prepared as an account of Government-sponsored work. Neither the United States, nor the National Aeronautics and Space Administration (NASA), nor any person acting on behalf of NASA:

- (A) Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately-owned rights; or
- (B) Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method or process disclosed in this report.

As used above, "person acting on behalf of NASA" includes any employee or contractor of NASA, or employee of such contractor, to the extent that such employee or contractor of NASA, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with NASA, or his employment with such contractor.

Requests for copies of this report should be referred to:

NASA Scientific and Technical Information Facility
P.O. Box 33
College Park, Maryland, 20740

1136

4 SEMIANNUAL MATERIALS REPORT

3 SNAP-8 MATERIALS REPORT ~~FOR~~ JANUARY - JUNE 1966
(By H. Derow and B. E. Farwell)

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

7 July 1966 10 CV

25 CONTRACT NAS 5-417 - 1

Technical Management
NASA Lewis Research Center
Cleveland, Ohio
SNAP-8 Project Office
H. O. Slone, Manager

Von Karman Center 5
AEROJET-GENERAL CORPORATION
Azusa, California 2

ABSTRACT

Investigations were conducted in support of the design, fabrication and development testing of various SNAP-8 components and system loops.

Operation of a Hg forced-convection corrosion loop was conducted with the aim of evaluating the corrosion resistance of 9Cr-1Mo alloy steel (the reference Hg containment material) for 10,000 hour service.

Data were developed in a continued program to evaluate the effect of the SNAP-8 turbine operating environment on Stellite 6B (the reference aerodynamic material).

FOREWORD

The ultimate objective of the SNAP-8 Program is to design and develop a 35-kw electrical generating system for use in various space missions. The power source will be a nuclear reactor furnished by the Atomic Energy Commission. The SNAP-8 system uses a eutectic mixture of sodium and potassium (NaK) as the reactor coolant and operates on a Rankine cycle, with mercury as the working fluid for the turbogenerator. The SNAP-8 system will be launched from a ground base and will be capable of unattended full-power operation for a minimum of 10,000 hours. After the system is placed in orbit, activation and shutdown may be accomplished by ground command.

The Materials Program was under the management of H. Derow, Head, Materials Section, Technical Support Department, SNAP-8 Division, Von Karman Center. Part of the work on this contract was performed at Aerojet-General Nucleonics under the direction of S. Nakazato, Liquid Metals Technology Department. The following engineers contributed to the various programs:

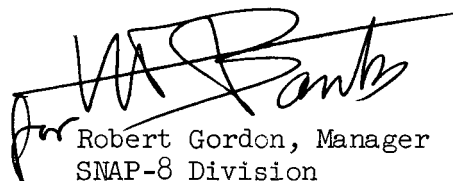
At Von Karman Center - H. Bleil and F. Cassidy,

At Aerojet-General Nucleonics - B. E. Farwell, A. Herdt, J. Ralphs, M. Wong, D. Yee, E. Johnson, H. Boarman and L. Kimura.

The suggestions and guidance of W. Titus, Metallurgy Department, Aerojet-General Nucleonics; and P. Stone, SNAP-8 Project Office, Lewis Research Center, NASA, are gratefully acknowledged.

This Semiannual Materials Report is submitted in partial fulfillment of Contract No. NAS 5-417. It covers the period from 1 January through 31 June 1966.

Approved:



Robert Gordon, Manager
SNAP-8 Division
Aerojet-General Corporation

CONTENTS

	<u>Page</u>
Abbreviations and Terminology _____	viii
Summary _____	x
PCS-1 Phase IV Step 2 _____	x
LNL-3 Post-Test Evaluation _____	xi
General Test Operations Support _____	xi
Turbine-Alternator Assembly _____	xi
Boiler Evaluation _____	xiii
Corrosion Loop Program _____	xiii
I. PCS-1 PHASE IV STEP 2 _____	1
A. NaK Primary Loop _____	1
B. Mercury Loop _____	3
C. L/C Loop _____	10
II. LNL-3 POST TEST EVALUATION _____	11
A. Evaluation Results _____	11
III. GENERAL TEST OPERATIONS SUPPORT _____	13
A. NaK Reclamation _____	13
B. Decontamination of Mercury Pressure Transducers _____	13
C. Specifications _____	13
IV. TURBINE-ALTERNATOR ASSEMBLY _____	15
A. Alternator Seal Weld Evaluation _____	15
B. Turbine _____	18

CONTENTS (cont.)

	<u>Page</u>
V. EVALUATION OF -1 BOILER (P/N 092020-1F, S/N A-1) _____	25
A. Operating History _____	25
B. Hg Inlet Plug _____	26
C. Twisted Ribbon Turbulator _____	26
D. Extrapolated Boiler Tube Life _____	27
VI. CORROSION LOOP PROGRAM _____	31
A. Corrosion Loop 4 Boiler Evaluation _____	32
B. Parametric Analysis of Mercury Corrosion _____	34
C. Mercury Wetting Tests _____	40
D. Mass-Transfer Tests - Corrosion Loop 4 _____	43
E. Corrosion Mechanism Loop -1 (CML-1) _____	46
References _____	54
	<u>Table</u>
NaK Gas-Fired Heater Wall Thickness Analysis Summary _____	1
Organics Analysis of Hg Dump-Tank Samples from PCS-1 Phase IV Step 2 _____	2
Fluids Analysis for Na, K, and Hg _____	3
Mix-4P3E Sample Analysis - PCS-1 Phase IV _____	4
Chemical Analysis of Filings from SNAP-8 Alternator Weld Areas After 140 Hours of Operation in PCS-1 Phase IV Step 2 Tests _____	5
Alternator Seal Weld Configurations and Results of Chemical Spot Tests of Welds _____	6
SNAP-8 -1 Boiler Performance History in RPL-2 _____	7
Depth of Surface Defects on Bar and Wire of Hg Inlet Plug from -1 Boiler _____	8

CONTENTS (cont.)

	<u>Table</u>
Effect of Geometry on the Performance Characteristics in the Preheat Region of the SNAP-8 Boiler _____	9
Effect of the Number of Channels per Tube on the Performance Characteristics in the Preheat Region of the SNAP-8 Boiler at a Constant Mass Velocity (G) _____	10
Effect of Thread Height and Pitch on the Performance Characteristics in the Preheat Section of the SNAP-8 Boiler _____	11
Summary of Capsule Wetting Tests _____	12
Summary of Removable Straight Sections - 4A Boiler _____	13
Geometric and Performance Parameters for the CML-1 Test Section _____	14
	<u>Figure</u>
Typical Appearance of ID of Pressure Tap from Hg Vapor Line of PCS-1 Phase IV After Step 2 Operation _____	1
Microstructure of Specimens Removed from LNL-3 After 3130 Hours of Isothermal System Operation at Between 1125 and 1175°F _____	2
Rockwell Hardness Traverse from Inside Pipe Surface of LNL-3 Pipe Specimens Removed After 3130 hours of Isothermal System Operation at Between 1125 and 1175°F (Hardness Converted from Knoop Microhardness Reading Taken with a 100-mg Load) _____	3
Schematic of System for NaK Purification by Filtration _____	4
Qualitative Hardness Test Results on SNAP-8 Alternator (S/N 481489) After 140 Hours of Operation in PCS-1 Phase IV Step 2 (Higher Numerical Values Indicate Greater Hardness) _____	5
S-816 Cobalt-Base Alloy After an Exposure of 2700 Hours at 1200°F. In the bottom Photomicrograph, the Darkened Islands are Carbides, and the Small Undarkened Islands May be Sigma Phase _____	6
Simulated TA Interstage Pressure Tap Braze Joint After 137 hours at 100°F in Air _____	7
SNAP-8 Tube-in-Shell Boiler _____	8
Hg Corrosion Effects on Core and Wire of Hg Inlet Flow Restriction Plug which Produced a Hg Velocity of 0.25 fps - Plug Removed from Tube Coil O-4 of -1 Boiler After 1415 hours of Operation in RPL-2 _____	9

CONTENTS (cont.)

	<u>Figure</u>
Ribbon Turbulator from Various Sections of Tube Coil 0-4 of -1 Boiler After 1415 Hours of Operation in RPL-2 _____	10
Corrosion Loop 3 1/19-Scale Boiler Test (Time: 4400 hours) _____	11
Corrosion Loop 4 1/19-Scale Boiler Test (Time: 2500 hours) _____	12
RPL-2 Full-Scale Tube-in-Shell Boiler Test (Time: 1425 hours) _____	13
Typical NaK Temperature Profiles and Pit Depths CL 4 Boiler _____	14
Microstructure of the NaK Side of the 9Cr-1Mo Tubing - CL 4 Boiler _____	15
Microstructure of the Mercury Side of the 9Cr-1Mo Tubing - CL 4 Boiler Coiled Section _____	16
CL 4 Boiler Inlet Section _____	17
Axial Distribution of Variables, Base Case _____	18
Effect of Thread Pitch and Height on the Maximum Corrosion Rate _____	19
Effect of Thread Pitch and Height on the Total Corrosion _____	20
Effect of Mass Velocity (G) on the Maximum Corrosion Rate _____	21
Schematic of Mercury Wetting Apparatus _____	22
Mercury Wetting Apparatus _____	23
Schematic of the 4A Boiler _____	24
4A-2 Test Section _____	25
4A-3 Test Section _____	26
4A-1 Test Section _____	27
4A-4 Test Section _____	28
CML-1 P & I Diagram _____	29
CML-1 Test Section _____	30
CML-1 Test Section Corrosion Performance (9M Samples) _____	31

ABBREVIATIONS AND TERMINOLOGY

Abbreviations commonly used in the SNAP-8 Program are defined below.

A	Angstrom units
AGN	Aerojet-General Nucleonics
ASTM	American Society for Testing Materials
BCC	Body-centered cubic microstructure
CL-4	Corrosion loop 4
CML-1	Corrosion mechanism loop 1
CTL-2	Component Test Loop 2
FCC	Face-centered cubic microstructure
HCP	Hexagonal close packed microstructure
Hg	Mercury
ID	Inner diameter
IR	Infrared
L/C	Lubricant-coolant
LNL	Liquid NaK loop
Mix-4P3E	Bis(mix-phenoxyphenyl)ether, a mixture of six possible isomers of bis(phenoxyphenyl)ether, considered as a lubricant-coolant for the PCS
NaK	Eutectic mixture of sodium and potassium
NPS	Nuclear power system
OD	Outer diameter
ORNL	Oak Ridge National Laboratories
PCS	Power conversion system
P/N	Part number
PMA	Pump motor assembly
PNL	Primary NaK loop
Rb	Rubidium
R _B	Rockwell B (hardness)
R _C	Rockwell C (hardness)

ABBREVIATIONS AND TERMINOLOGY (cont.)

RPL-2	Rated Power Loop 2
SL-1	System Loop Test Facility No. 1
S/N	Serial number
SNAP	Systems for Nuclear Auxiliary Power
SS	Stainless Steel
TAA	Turbine-alternator assembly
TRA	Transformer-reactor assembly
T-T	Tube-in-Tube
VKC	Von Karman Center
XRD	X-ray diffraction

SUMMARY

Materials work during the first half of the 1966 calendar year provided data to guide the selection of materials for SNAP-8 system components. Metallurgical assistance was also provided in the design, development, fabrication, and testing of the system.

PCS-1 Phase IV Step 2

The wall thickness of the NaK containment pipes in the two gas-fired NaK heaters of the PCS-1 Phase IV test loop was determined by ultrasonic pulse-echo technique. Estimated remaining heater life ranged from 6,200 to 12,600 hours. General wall thinning occurred since the last previous inspection in June, 1965 (heaters had operated 970 hours between inspections). The continued use of the heaters for Step 3 (of the PCS-1, Phase IV sequence) testing, with reinspection after an additional 1000 hours of operation, was recommended.

A ductile failure of a liquid-level probe from the PCS-1 Phase IV primary NaK expansion tank during Step 1 operation occurred because the probe was stressed to its ultimate strength probably during insertion and/or an unsuccessful attempt to remove it from the tank. No material deficiencies were detected. Metallic fragments did not separate from the probe during failure; however, it is estimated that 0.0007 cubic inch of the magnesium oxide insulation separated from the probe. No adverse effects on loop operation should result from the separation of the insulation.

NaK contamination of the Hg loop inventory was apparently caused by external sources, probably by the use of previously contaminated NaK-exposed TSE components. There was no apparent cross-loop leakage and the NaK quantity was so low as to be considered not detrimental to continued use of the Hg inventory.

Arcing of an electrical line heater during a startup produced two holes in the 316 SS Hg vapor line. Visual examination revealed that the influx of air into the hot line through the holes had oxidized the inside surface of the line. The amount of oxidation was apparently dependent on the varying temperature of the metal along its length. Some areas contained a loose flaky oxide scale while others had a tenacious oxide coating. Metallographic examination of a hot-spot area remote from the arcing location and in a cooler area of the loop indicated that the material was satisfactory for continued use. No indications of grain growth, incipient melting, or subsurface or intergranular oxidation were evident.

Corrosion occurred in several 316 SS pressure taps that had operated in the Hg-vapor section of the Hg loop. The corrosion rate is estimated at 10.6 to 14.2 mils per 1000 hours. Extrapolated anticipated total life is 2500 to 3350 hours. The material was exposed to continuous Hg vapor refluxing and appeared to be insufficiently resistant to Hg corrosion. A change to 9Cr-1Mo steel was recommended.

The failure of two 316 SS thermowells in the Hg vapor line was attributed to the incompatibility of indium, a heat-transfer medium used inside the well, and 316 SS, the well material. The mode of failure was localized ID wall thinning and eventual wall penetration. It was recommended that the use of indium in 316 SS thermowells be discontinued and that other materials or methods to provide the required sensitivity and response in temperature measurement be investigated.

Analysis of mix-4P3E samples removed from the L/C loop after the 14 February shutdown indicated that the fluid was acceptable for continued use. The spectrogram, isomer distribution, specific gravity, refraction index and viscosity of a dump-tank sample were within the tolerances specified for new material in the mix-4P3E procurement specification (AGC-10320).

LNL-3 Post-Test Evaluation

No metallic mass-transfer deposits were found after this NaK system had operated isothermally for 2655 hours at temperatures between 1125 and 1175°F, and an oxygen content of less than 30 ppm. Carbon migration occurred from the mild steel (0.020 in. maximum decarburization depth) to the 316 SS (0.010 in. maximum carburization depth) in the system. The amount of transfer was not sufficient to deleteriously affect loop operation should it be used for additional NaK PMA tests.

General Test Operations Support

A filtration procedure was developed and successfully used to process approximately 1800 lb of previously used NaK which had been removed periodically from various test systems. The processing produced NaK that conforms to the SNAP-8 procurement specification (AGC 10340).

Turbine-Alternator Assembly

Variations in the weld configuration from unit to unit were found in the seal welds between the HY-80 (low alloy steel) trunnion ring and the mild steel case on five GE fabricated prototype alternators (P/N 094069). The type and amount of weld filler metal used to complete this joint also appeared to have been varied. Chemical analyses of filings from the alternator seal weld that cracked in service (S/N 481489) indicated that alloying due to melting of the parent metals produced a weld composition which is hardenable. Weld hardening appears to be accentuated by the chilling effect of the massive HY-80 trunnion boss. A probable major factor contributing to the failure of this unit was this hardened, and consequently less ductile, metal structure. Chemical spot tests of the other four alternators, which have not been tested at AGC, indicated that the seal welds in these units might also be subject to seal-weld cracking. The potential for cracking appeared to be a function of the weld configuration and weld filler metal used in each unit. Since stress relieving (a weld-tempering heat treatment) of these units was not practical, it was recommended that a multiple-pass overlay weld be deposited on the present seal weld using Inco A filler, a metal that is nonhardenable and

ductile. It was recommended that for future units, the seal-weld design, materials selection, and fabrication procedure should be reviewed to avoid non-ductile welds.

Transformation of Stellite 6B tension and impact test specimens from FCC to HCP crystalline structure for the determination of mechanical properties was unsuccessful using the previously established standard thermal treatment (1650°F for 4 hours followed by 1250°F for 48 hours). The hardness increased only slightly (from R_C 37 to 39) and the HCP content was well below the required minimum (30% and 80% respectively). Preliminary tests indicated a soak temperature of 1450°F produces the most rapid transformation. After exposure of up to 1100 hours, all of the impact specimens were satisfactorily transformed and final specimen machining was started. The tensile specimens have been exposed for as long as 800 hours without acceptable results. Exposure is continuing.

Exposure of Stellite 6B and S-816 (at 1065 and 1200°F respectively) specimens was continued to evaluate the metallurgical stability of these two turbine aerodynamic cobalt-base materials. The Stellite 6B was treated by salt-bath quenching to 1000°F from the solution annealing temperature, 2250°F, to stabilize the FCC structure. The two materials have proven stable at 1065°F after 3955 hours (Stellite 6B) and 4275 hours (S-816) exposure. The Stellite 6B specimen exposed at 1200°F transformed to the HCP structure after 1710 hours. With the currently established metallurgical knowledge it appears that Stellite 6B transformation from FCC to HCP cannot be prevented at 1200°F. S-816, another cobalt-base alloy, exhibited thermal stability after 4275 hours at 1200°F.

PH 15-7 Mo, a precipitation hardenable stainless steel used for the turbine-alternator assembly second-stage shroud, in the TH 1050 condition, was found to be metallurgically unstable at 1065°F. The material is not recommended for use in the SNAP-8 turbine. Although stable alloys should be substituted for the PH 15-7 Mo this material must be used in the interim. Therefore, a preassembly thermal treatment was developed to produce metallurgical stability at the turbine operating temperature 1065°F. This treatment incorporates a 50-hour soak at 1065°F of the material after it has been heat treated to the TH 1050 condition, but before final machining has been performed.

An Inconel 718 (nickel base alloy) turbine assembly retainer spring was evaluated for Hg corrosion effects after 117 hours of operation in the S/N 6/1 turbine. A plated surface layer (pure nickel over iron which was plated directly on the spring) appeared unaffected by the Hg exposure indicating that nickel components may be usable in the first and second stages of the turbine without catastrophic Hg corrosion.

A brazed joint developed for connecting the turbine assembly interstage pressure taps was found to be metallurgically stable after exposure of samples at 1000°F (the maximum operating temperature) in air for approximately 2000 hours. No detrimental reaction occurred between the Easy Flo braze alloy and either the mild steel pressure tap or 9Cr-1Mo steel turbine case representing the S/N 5/2 turbine configuration. The joint is considered acceptable for turbine tests of at least 2000 hours duration.

The remelt temperature of a brazed joint consisting of Easy Flo braze alloy, 316 SS pressure tap and 9Cr-1Mo steel turbine case, representing the S/N 2/3 turbine configuration, was established as 1145°F, and rupture of the joint required 47 psi (lap shear strength). Thus, it appears that removal of the taps is practical should it prove necessary during S/N 2/3 turbine assembly testing.

Boiler Evaluation

The Hg inlet plug from the -1 boiler (P/N 092020-1F, S/N A-1) was examined as part of the continued evaluation after the boiler operated for 1425 hours in RPL-2. Both the mild steel rod and mild steel wire which comprise the plug were attacked. The maximum depth was 4.2 mils, measured 1 ft-4 in. from the Hg inlet. Both elements were attacked at the rod-wire interface indicating Hg bypass flow in the axial direction rather than the helical path formed by the wire.

The twisted ribbon turbulator, located downstream of the plug to maintain vapor vortex flow, had been stress relieved of the cold work strains imposed during twisting. The boiler operating temperature, between 1100 and 1300°F produced the effect. The stress relieving caused local buckling of the ribbon. The buckled areas acted as corrosion product traps, containing large accumulations of porous, dendritic mass-transfer buildup. The maximum buildup was 3.3 mils at 46 ft-8 in. from the Hg inlet.

It is estimated that a boiler of this type would have operated approximately 11,000 hours, based on penetration attack being the life limiting mechanism. This estimate is based on the conservative assumptions of a 0.200-in. thick wall in the area of maximum attack rather than the current 0.090-in. wall thickness; and the limit of penetration defining boiler life is through only the added thickness, 0.110 in.

Corrosion Loop Program

A parametric analysis of the corrosion in the preheat section of the boiler was made to try to determine the design parameters that can be modified to reduce the boiler corrosion rate.

Mercury wetting capsule tests were started to provide a procedure that would assure complete local wetting of the mercury containment tube in the boiler.

The original mercury boiler in corrosion loop 4 (CTL-4) was removed and a modified boiler, designated 4A, was installed. This boiler was designed so that the entire boiler inlet plug straight section can be removed and readily replaced. The first of four boiler inlet plug test sections was installed in the CL-4A boiler and operated to obtain mass-transfer and heat-transfer data.

Work was initiated on a single-phase liquid mercury loop designated corrosion mechanism loop 1 (CML-1). The objective of this loop is to determine the mechanism that controls the corrosion of 9Cr-1Mo in mercury.

I. PCS-1 PHASE IV STEP 2

This loop is being operated to perform a 10,000-hour test on a system containing all prototype SNAP-8 components with the exception of the nuclear reactor. The reactor is simulated by gas-fired NaK heaters. The NaK primary loop of this system has operated for approximately 4200 hours. For the first approximately 3000 hours, the system carried the designation RPL-2.

A. NaK PRIMARY LOOP

1. Gas-Fired NaK Heater Pipe Evaluation

Component and system tests at Aerojet-General use gas-fired NaK heaters to simulate the nuclear power system (NPS) concurrently being developed by Atomics International. Two elements of operation may be detrimental to the service life of the NaK containment piping in the heaters. First, the pipe wall thickness may be reduced by removal of internal wall material by mass transfer - especially accentuated in NaK loops operated with a high oxygen content. Second, oxidation of the external pipe surface may be caused by exposure to the heater flame and air. Cyclic loop operation, heating and cooling, can accentuate the oxidation rate due to spalling of otherwise protective oxide coating.

The gas-fired NaK heaters used in this system have experienced many thermal cycling restarts and operated for slightly less than 1000 hours with significant oxygen contamination following a primary loop massive NaK leak. Wall thickness measurements of the piping in two NaK heaters were made to ensure that oxidation and mass transfer had not unacceptably reduced the pipe wall thickness, and to estimate additional safe operating life.

The wall thickness of the heater NaK containment pipes was determined by the ultrasonic pulse echo technique. This repeated a similar inspection performed in June 1965 (Ref. 1). No definite pattern of thinning from the NaK inlet to the outlet was observed; however, a general wall thinning had occurred during the 970 hours of operation since the previous inspection. Additionally there is an obvious inconsistency in the results produced by the two approaches as well as in the comparison of the current estimates and those previously made in June 1965. These differences can only be ascribed to insufficient data and the necessity of making many assumptions. This is especially true of the June 1965 estimate when no base point was available. The original survey indicated an estimated remaining heater life of 9100 hours for heater 1,

and 11,700 hours for heater 2. A reestimate of anticipated remaining heater life was made using the current data, Table 1. The estimate was based on the wall thinning rate; however, this rate varied depending on the method of determination. Two approaches were taken. The first procedure was based on the wall thinning rate determined from the measured average wall thickness. The result was estimated lives of 12,600 hours for heater 1 and 7,200 hours for heater 2. The second method was based on the wall thinning rate determined from the measured minimum wall thickness. The result was estimated lives of approximately 6,400 hours for heater 1 and approximately 6,200 hours for heater 2.

Heater 2 exhibited a greater average wall thinning than heater 1, for reasons not apparent at present.

It is concluded that the heaters are safe for initiation of PCS-1 Phase IV Step 3 testing, and that another thickness determination should be performed at a convenient time after an additional 1000 hours of operation. The next survey during Step 3 testing should provide sufficient data to confirm the accuracy of the current estimate.

2. Expansion Tank Liquid Level Probe

Sheathed Chromel-Alumel thermocouples are employed in the primary NaK loop (PML) expansion tank to indicate the NaK liquid level. One of the probes had to be bent during installation. It apparently was damaged at this time because it never functioned. During Step 1 operations, an unsuccessful attempt was made to remove this probe for examination. After completion of the Step 1 testing, it was determined that mechanical failure of the probe had occurred and a section of it had fallen into the tank. Both ends of the failed probe were recovered and evaluated.

a. Evaluation Results

Failure had occurred adjacent to the area where the probe was welded to a gusset. The gusset is intended to stiffen the thermocouple against excessive flexing and distortion. Below this location the coiled thermocouple was relatively flexible. The ends of the separated sections matched sufficiently so that it may be presumed that metal fragments were not missing. The failed sheath area appeared to have retained ductility and to have undergone plastic flow, as indicated by reduced wall thickness. The thermocouple wires were bent almost to the point of contacting the sheath in the failed area. The wires were also necked down, indicating ductile failure. Approximately a 1/16-in. length of the magnesium oxide insulation was missing from each side of the failure (0.0007 in.³) and is presumed to have been introduced into the expansion tank.

Microscopic examination of a section through the failure confirmed that plastic flow had occurred. This was indicated by elongated grains at the failure edge, the presence of many slip planes in the grains at, and adjacent to, the failure, and by an increase in hardness

of both the sheath and the thermocouple wire produced by cold working during the failure. The micro-hardness of the AISI 316 SS sheath converted to Rockwell hardness was R_C 45-50 at the failure edge and R_C 22 in areas remote from the failure. The latter hardness is higher than would be anticipated for annealed material (R_B 90 max). However, this is due to the cold working which occurred during coiling of the probe. The cold-worked structure remained since the probe was not annealed after coiling. This presumption is supported by the appearance of slip planes in the grains remote from the failure.

No microstructural indications of corrosion by the NaK or of sensitization of the 316 SS were visible. This tends to indicate that the probe was probably not exposed to temperatures greater than 800°F in the expansion tank.

b. Discussion

It is evident from the results of the evaluation that the probe failed because stresses were applied which could not be elastically accommodated by the metallic materials of the probe. The reported difficulty in installation and removal of the probe was probably the major contributor to the failure. The location of the failure, at a stiffening point, is such that only plastic flow could produce significant movement. In producing the movement during the above incidents, the ultimate strength of the material was reached and failure occurred.

Based on free energies of formation the NaK will not decompose the MgO insulation. The particle size of the MgO which was introduced into the tank could be anywhere from 0.020 in. down to fine powder depending on the extent of fragmentation that occurred. However, this particulate matter should not cause problems with loop or component operation since the filters will trap any large particles and those that pass through the filter will be no larger than the maximum size corrosion and mass-transfer products which pass through the filter.

As a result of the investigation it was recommended that the fittings for metallic components should be designed so that the fit of parts is sufficient to ensure against unwanted plastic deformation during assembly; and, the thermocouple design should be reviewed to eliminate the area of inflexibility at the reinforcing gusset.

B. MERCURY LOOP

1. Mercury Analysis

a. Organics

During this report period, PCS-1 Phase IV Step 2 testing was started and completed. It had previously been demonstrated on the SNAP-8 program that organic contamination can cause poor boiler performance.

Organic contamination of the Hg loop is possible by cross transfer of the mix-4P3E in the turbine assembly and Hg pump-motor assembly (PMA) space seals, and by back-streaming of vacuum pump oil during evacuation of the loop. Numerous Hg-loop shutdowns occurred for various reasons during Step 2 operations. Therefore, to determine if organic contamination had occurred, Hg samples were taken whenever practical after shutdowns and analyzed for the presence of organics. While some organic content was found in the Hg dump tanks after the shutdowns, it was generally at such a low level that it was considered insufficient to cause difficulties with boiler performance. This was confirmed by good boiler performance each time the loop was restarted. After the second to the last shutdown, no samples were taken because of imminent restart. The restart indicated the boiler was deconditioned and the loop was shut down and dump tank samples were taken. Analysis of these samples indicated that relatively large quantities of an organic had entered the boiler and had caused the deconditioning observed. Step 2 operations were terminated with most of the desired test data obtained. The Hg containment portions of the loop were cleaned to remove organic contamination prior to the initiation of Step 3 testing.

(1) Analyses Prior to Step 2 Operation

Prior to Step 2 operation of PCS-1 Phase IV, analysis of samples taken from the main Hg dump tank was performed. The mercury itself was not contaminated with organics or alkaline material at a detection limit of 0.01 ppm by weight (alkalinity calculated as potassium for alkaline metals), and the Hg surface was also free of organics. The emergency dump tank was contaminated with an aliphatic hydrocarbon. The organic present in the emergency dump tank is isolated from the loop, and so it was recommended that Step 2 operations be initiated with the Hg inventory and dump tanks as they were without cleaning.

(2) Analysis of Samples Taken During Shutdowns During Step 2 Operation

The results of the organics analyses performed on samples of Hg taken during the calendar time period of Step 2 operations are listed in Table 2. These results indicate that, except for the final samples, a low level of contamination of the loop by mix-4P3E and an aliphatic hydrocarbon occurred. The samples taken on 16 March 1966 indicated the presence of an ester and/or possibly carbonyl. The ester functional group $R-C(=O)-R'$ (where R and R' are the same or different organic groups) absorbs infrared radiation at 5.8 microns and from 7.9 to 8.6 microns of the infrared spectra. The carbonyl functional group, $R-C(=O)-R'$ absorbs infrared radiation at 5.8 microns. Thus, if carbonyl is present with an ester, it may not be possible to distinguish its presence. The IR trace of this organic was identical to that of Octoil, the vacuum pump oil employed in the Veeco leak detector. Analysis for the presence of the aliphatic hydrocarbon was not practical since strong infrared absorptions occur at the same wave lengths as for the Octoil-type organic detected. Aliphatic hydrocarbons (i.e., open-chain hydrogen-carbon compounds vs aromatic, or ring-chain hydrocarbons) refer here to those hydrocarbons, containing only hydrogen and carbon, that absorb infrared radiation in the 3.4 micron (CH_2 and CH_3 radicals), 6.9 micron (CH_2 radical) and 7.3 micron (CH_3 radical) wavelengths, similar to vacuum pump oils like Duoseal. Octoil contains CH_2 and CH_3 radicals in addition to the

ester functional group and, thus, absorbs infrared radiation in the aliphatic hydrocarbon wavelength as well as in the ester wavelengths. Therefore the presence of an aliphatic hydrocarbon, if any was present, was completely masked.

The only apparent potential source of Octoil is the vacuum pump in the leak detector; it was recommended that extremely close care be taken in maintaining the cold traps and that other possible sources of this organic contamination be investigated.

After each of these analyses was performed, it was recommended that Hg operation be continued without loop cleaning. In each case, the restart of the system and good boiler performance confirmed the validity of the recommendation. It does not appear that organic contamination in the amounts observed prior to the final analysis caused degradation of boiler performance within the limits of detectability by the test instrumentation.

(3) Analysis of Hg Samples from Dump Tanks After Step 2 Completion

After a shutdown of loop operation on 12 April 1966, no Hg samples were taken because of imminent restart. The restart indicated that the boiler was deconditioned. The loop was shut down and Hg samples from the dump tanks were obtained and analyzed. Although only the usual small quantity of an aliphatic hydrocarbon was detected in the main dump tank, considerably higher quantities of organics were detected in the emergency dump tank, and the Hg visually appeared to be contaminated with a black residue. The analysis of the mercury indicated it contained 6.4 ppm of mix-4P3E and 5.1 ppm of an aliphatic hydrocarbon. The IR trace also indicated that some organic oxidation had occurred as evidenced by the presence of double bonded oxygen and carbon atoms. Further evidence that oxidation and/or decomposition of an organic had occurred was indicated by the presence of 5.35% carbon in the black residue from the Hg for the emergency tank. These analysis indicated that mix-4P3E had entered the boiler in significant quantities and thermally decomposed, probably leaving a heat-transfer-inhibiting film on the boiler tube wall. Therefore, it was recommended that the boiler be cleaned prior to the continuation of operations. Since most of the desired test data had been obtained, Step 2 operations were terminated and the Hg system was chemically cleaned to remove organic contamination.

(4) Analysis of Hg Drained from the Main Dump Tank

Before the main dump tank was cleaned, the Hg inventory was siphoned from the bottom of the tank into polyethylene bottles. The first twenty bottles siphoned from the tank visually appeared clean. The last three partial bottles obtained contained a surface residue.

Mercury from the first twenty 30-lb polyethylene bottles removed from the main dump tank met the requirements of Aerojet's Development Material Specification, Heat Transfer Grade Mercury (AGC 10327). Therefore, it was recommended that this Hg be reused in Step 3 operations without re-processing.

The Hg in these bottles appeared clean with no visible surface residue. Two composite samples were analyzed each representing ten of the bottles. The following results were obtained:

<u>Analysis for:</u>	<u>Composite 1</u>	<u>Composite 2</u>	<u>AGC 10327</u>
Nonvolatile residue	0.5 ppm	0.7 ppm	6.0 ppm max.
Na	0.023 ppm	0.025 ppm	not required
K	0.070 ppm	0.071 ppm	not required
Aliphatic hydrocarbon	0.05 ppm	<0.05 ppm	not required
Mix-4P3E	ND	ND	not required

ND = not detected at a detectability limit of 0.005 ppm

As a control, a composite sample representing seven polyethylene bottles of reprocessed Hg was also analyzed for organics. This analysis indicated that the reprocessed Hg contained 0.10 ppm aliphatic hydrocarbon, but no mix-4P3E was detected. Since all of the Hg analyzed had been stored in polyethylene bottles, the possibility of picking up an aliphatic hydrocarbon from the bottles is being investigated. Also, the Aerojet specification (AGC 10327) will be modified to include a requirement for specifying organic content in the Hg.

The last three bottles of Hg removed from the dump tank included some of the surface material that had floated on the Hg. Undoubtedly some small fraction of the residue was retained on the walls of the tank as the Hg was drained. Therefore, an exact analysis of the ratio of floating residue to Hg, as it existed when the dump tank was full, could not be made. If all of the residue in the tank had been collected, the amount of extraneous material present would have been slightly higher than indicated in Table 2. However, it is presumed that the residue which was obtained represents essentially all of the total residue in the tank and the quantity remaining on the wall may be neglected. Observations made on the effluent collected from a previous dump tank which was subsequently cleaned after removal of the contaminated Hg inventory support this presumption. The results indicate that both an aliphatic hydrocarbon and mix-4P3E were present in small amounts in the surface residue of the dump tank. The concentration measured was not so high that a recommendation not to use this Hg would have been made if the dump tank had not been emptied. However, since this Hg had been separated from the bulk of the Hg inventory from the main dump tank, it will not be used for Step 3 operations.

b. Alkali Metal Contamination

The presence of both sodium and potassium in small quantities and in eutectic ratio was detected by flame emission spectrographic analysis in Hg samples from both the emergency and the main dump tanks (taken after completion of Step 2 testing). Both elements were also detected in Hg removed from the emergency dump tank on 1 February 1966. The ratio was not eutectic; however, this may have been influenced by preferential oxidation or the sampling technique. Sodium was detected in reprocessed Hg and in mix-4P3E from the L/C dump tank, but no potassium was detected.

The amount detected did not require Hg inventory replacement; however, to determine if a cross transfer of fluids between

loops had occurred through small undetected leaks, samples of NaK from the primary and the heat rejection loops, as well as from unused vendor supplied NaK, were analyzed for Hg content. All of the NaK samples contained approximately the same small quantity of Hg with the unused material containing the greatest amount (Table 3). It was concluded that cross transfer of the loop fluids had not occurred, and that the most probable source of the Na and K in the Hg from the loop was the possible use of transducers or valves in the Hg loop which had previously been employed in a NaK loop. Another possible source of NaK in the Hg is its introduction when the condenser was cut into and rewelded during RPL-2 operation. At that time, during the repair work, it is known that the NaK side of the condenser was cut into and that difficulty was experienced in the repair welding of the Hg side due to the presence of NaK at the weld area.

2. Vapor Line Failure

Failure of an electrical line heater during a March 1966 startup produced two holes in the 316 SS Hg vapor line by electrical arcing. The internal surface of the penetrated section of the Hg-loop vapor line evidenced significant air intrusion into the loop while the pipe was at temperature prior to Hg injection. Loose and adherent internal surface scale was found indicating that some portions of the vapor line were at a temperature approximating 1600°F or above. The turbine inlet filter was examined; no sign of detrimental effect to the filter element was found. The loose residue which may have been present in the Hg vapor portion of the loop was removed by steam cleaning of the vapor line since the friable particulate matter could cause condenser-tube plugging during subsequent shutdowns.

The 9Cr-1Mo boiler tube was at 1300°F at the time because of presence of heated NaK on the PNL side. The presence of scale in the Hg vapor line indicates that the boiler tube was probably exposed to air long enough when the vapor wall penetration occurred to form an oxide surface film on the Hg side.

Two tests completed previously were considered in the decision on action required prior to system restart. In the first test, air was flowed through the Hg tube of a single tube-in-tube boiler test section while the tube was at 1300°F. After one hour of air flow, a normal boiler start was successful; i.e., immediate conditioning was achieved. In the second test, 9Cr-1Mo specimens were exposed to air at 1300°F to test oxidation rates. Times in excess of 168 hours at temperature resulted in only minor specimen weight changes, 0.03 g/in.², and there was no loose surface scale.

From all information available, it was concluded that the Hg tube of the system boiler probably has an oxide surface film as a result of the 316 SS Hg vapor line wall penetration. This film is probably very adherent and would visually appear as surface discoloration rather than flaky, easily dislodged scale. Previous test evidence indicates that this film may not be detrimental to boiler conditioning.

It was recommended that the Hg vapor line should be steam cleaned and that restart of the system should be attempted without boiler chemical cleaning. This was done and the boiler conditioned satisfactorily.

3. Instrumentation Evaluation

a. Pressure Taps

(1) Wall Deterioration

Three 316 SS pressure taps from the Hg vapor line, within seven feet of the boiler outlet (PCS-1 Phase IV), were examined to determine whether or not Hg attack of the material had occurred during the 302 hours of Step 2 Hg operation. These taps had been installed with a five degree downward slope away from the vapor line (Figure 1a). This configuration would permit Hg condensing reflux. The examination revealed that thinning of the tap walls had occurred at the hot end adjacent to the loop Hg containment pipe. The thinning tapered off within approximately three inches of the hot end, and the tube remained uniformly thick for the rest of the length to the transducer attachment (cold) end. Based on the uniform cold-end wall thickness, maximum wall thinning, ranging from 3.2 to 4.3 mils was observed. This represents an estimated corrosion rate range of 10.6 to 14.2 mils per 1000 hours. This rate, if continued, would result in the wall reaching the 0.0125-in. minimum boiler code allowable wall at 1300°F and 265 psi in 2500 to 3350 hours. These figures compare to a previously examined tap which exhibited an 11-mil wall thinning in 1430 hours, and an estimated safe operational life of 4800 hours, Reference 2. Metallographic examination of the taps indicated the wall thinning was due to general dissolution corrosion; however, a minor amount of pitting corrosion to a depth of approximately 0.5 mil also was observed. It was concluded that 316 SS pressure taps would not be suitable for long-term applications where Hg condensing reflux occurs. Therefore, it was recommended that these taps be replaced with 9Cr-1Mo alloy steel.

(2) Residue Analysis

The vapor line including the pressure taps had been cleaned in the system (in accordance with Aerojet specification AGC-10319/8) before being examined. Although the vapor line itself visually appeared to be clean, the taps did contain a general layer of residue adjacent to the wall (Figure 1b), which was removed with a bristle brush and nitric acid solution before the wall thicknesses were determined. Analysis of this nonmagnetic residue indicated that it contained 44% Fe, 9% Ni, 8% Cr, 1.6% Mo and 2.3% Mn. It is presumed that this residue collected in the taps during loop operation and floated on the Hg which condensed in the taps. During shutdown and startup of the loop, the liquid Hg in the taps evaporated leaving behind a relatively uniform coating of the residue on the ID of the taps. X-ray diffraction powder patterns of this material indicated it was amorphous. It was concluded that this material was composed of corrosion-oxidation products, the primary constituent being $\text{Fe}_2\text{O}_3 \cdot \text{XH}_2\text{O}$. It was also concluded that hydration of the oxide occurred during the cleaning of the system with aqueous solutions. Because of flow stagnation in the taps, the material was not removed during the cleaning operation.

In addition to the general residue, a tan-colored residue was present at the hot end of the pressure taps on the bottom side only. Debye Sherer patterns and chemical analysis indicated that this material was Na_2CO_3 . It is probable that this residue is a result of cleaning solution retention in the taps after draining. Although the caps placed on the taps after removal of the transducers were loosened as part of the cleaning operation to drain the solutions, an ID weld ridge close to the vapor line caused the retention of solution close to the vapor line. The water was evaporated during the drying operation leaving behind sodium hydroxide which reacted with CO_2 from the air to form the sodium carbonate. Since all of the vapor line pressure taps were replaced after PCS-1 Phase IV Step 2 operations, no detrimental effects of these residues will result for Step 3. It was recommended that, during subsequent loops cleaning operations, the solutions be flushed from the pressure taps with water prior to drying.

b. Thermowells

Thermowells are used in the PCS-1 Phase IV system to provide greater accuracy and more rapid response than was found to exist with surface-contact-type couples previously used. Thermocouples are inserted into the wells and the low melting (313°F), low-vapor-pressure metal, indium, is placed in the well to provide a good heat transfer medium between the well wall and the couple. Tests had shown that indium was compatible with 316 SS. Although the tests simulated the temperature of the system, they did not simulate the liquid metal flow or thermal gradients that exist in the loops. The present investigation (Ref. 2), represents a more realistic evaluation of the compatibility of 316 SS and indium as it has been used in SNAP-8 loops.

After 143 hours of Hg loop operation, a vapor line thermowell (T213) failed. The failure was discovered when the vacuum in the Hg loop deteriorated as the loop was being evacuated prior to Hg injection. This well was capped off and operation of the loop continued. After completion of Step 2 operations, the Hg boiler and the vapor line were chemically cleaned. Subsequently, a section of the 316 SS Hg vapor line, from approximately seven feet downstream of the boiler to the boiler outlet, was removed from the loop for examination. This section of line, including the five 316 SS thermowells it contained, had been exposed to Hg for 302 hours of operation. For comparison, an 18-in. length of the 316 SS primary NaK loop piping adjacent to the boiler NaK inlet and containing three 316 SS thermowells, all of which had been exposed to 2422 hours of PNL operation, was also removed for examination.

The five thermowells from the first seven feet of the Hg-vapor line, and the three thermowells from the PNL near the boiler NaK inlet were examined. It was determined that, under the SNAP-8 operating conditions in these areas, indium is not compatible with 316 SS and that diffusion, alloying, and the probable formation of brittle intermetallic compounds occurs. Failure of two of the wells in the Hg vapor line was attributed to this incompatibility which led to localized wall thinning from the ID of the wells. Although the complete mechanism leading to

failure of the wells is obscure, it was determined that indium contributes very significantly to the problem. Therefore, it was recommended that the use of indium in the thermowells be discontinued.

C. L/C LOOP

1. Mix-4P3E Sample Analysis

Samples were recovered from various loop locations after the 14 February system shutdown. These were analyzed (Table 4) to determine L/C loop operational effects on fluid properties. Analysis of a sample removed from the dump tank indicated that this fluid was satisfactory for continued use.

All samples exhibited typical mix-4P3E spectrograms. Two of the samples (one from the alternator drain trap and the other from the vacuum pump trap) also contained small amounts of vacuum-pump oil contamination, due to the position of these traps relative to the vacuum pumps in the system.

Specific gravity, refractive index, and viscosity of a dump tank sample were within the tolerances specified for new material in the SNAP-8 procurement specification (AGC-10320). The refractive index of the L/C vacuum pump trap was low, but not unacceptably so. The low value was probably due to the vacuum pump oil contamination of the sample. Vacuum pump oil is less dense, is not miscible with the mix-4P3E at room temperature and has a lower refractive index. The mix-4P3E sample was removed from the surface of the inventory and any vacuum pump oil which may have been present would have been difficult to separate completely.

Isomer distribution of the dump-tank sample was somewhat different from the control. Cause of the difference is uncertain, but it may have been the result of intermixing several different batches. The distribution is considered acceptable for continued use of the fluid.

2. Loop Cleaning

Mercury droplets were found in the L/C loop. Existence of mercury in the mix-4P3E could be detrimental to the L/C PMA, Hg PMA, alternator and the aluminum components of the transformer-reactor assembly (TRA). The total amount of contamination could not be determined. Therefore, two lines of the L/C loop were analyzed to determine the extent of mercury contamination. The return lubricant line from the Hg PMA bearing (surface area of approximately 3.6 ft², much of which was vertical) contained 45 g of finely divided mercury which was difficult to remove by degreasing. The decision was made to clean the complete L/C loop. The cleaning procedure involved removing of all the lines, degreasing to remove mix-4P3E and nitric acid pickling using the acid defined in Aerojet specification AGC 10319/10, Method 1.

II. LNL-3 POST TEST EVALUATION

The SNAP-8 liquid NaK test loop (LNL-3) was operated to provide information needed to evaluate the performance of the NaK PMA. This isothermal loop had operated approximately 3130 hours, 2655 hours at temperatures between 1125 and 1175°F. No oxygen content determinations were made during the initial low-temperature operating period. The oxygen content at the NaK during high-temperature operation was controlled below 30 ppm through a cold-trap purification system. Samples of the loop piping were removed for metallographic examination (Figure 2).

A. EVALUATION RESULTS

No metallic mass-transfer deposits were detected by visual or metallographic examination of the inside pipe surfaces of any samples. The outside pipe surfaces appeared oxidized as a result of air exposure during high-temperature loop operation. Metallographic examination and micro-hardness determinations revealed, however, that carbon migration, on the NaK side, from the mild steel portion of the system at the flow venturi to the 316 SS NaK containment pipe had occurred. Decarburization depth as determined by microhardness measurements (Figure 3) in the mild steel was approximately 0.020 in., carburization depth in the hot end of the 316 SS cold finger was approximately 0.010 in. Only minor hardening occurred in the 304 SS at the pump discharge and the 316 SS at the venturi inlet. It appeared that most of the effect of the transported carbon occurred in the stainless steel downstream of the venturi and upstream of the pump discharge. It is expected that, with continued loop operation under the same conditions and with the same materials, carbon migration would continue but at a reduced rate because the carbon must diffuse a greater distance to the surface of the mild steel before it can be transported by the NaK. It is also expected that a loss in ductility and impact strength of the stainless steel will occur as a result of the surface carburization, but that it should not deleteriously affect loop operation.

X-ray diffraction analysis of the stainless samples indicated that no ferrite had formed in the material during loop operation. It was concluded that the loop piping materials had not been deleteriously affected by the system operation and that continued operation of the loop would be satisfactory from a materials standpoint.

III. GENERAL TEST OPERATIONS SUPPORT

A. NaK RECLAMATION

A large inventory of used NaK had accumulated as a result of replacement of loop inventories. Generally, the replaced quantity had been in service for periods, which included high oxygen contamination, long enough to cast in doubt the NaK purity level and its acceptability for reuse. A study of alternatives indicated that the least expensive, most expeditious procedure for replacing contaminated NaK with certified (to the SNAP-8 NaK procurement specification AGC-10340) fluid was by filtration of the contaminated NaK to remove oxides and other solids, and recertification tests to ascertain acceptability of the composition. A total of 1788 lb of contaminated NaK was successfully processed.

A filtration system utilizing 2 filters in series was used (Figure 4). The contaminated NaK container is pressurized to push the contaminated NaK through a large filter containing York stainless-steel mesh. This acts as a depth filter and removes the greatest portion of the contamination. The NaK then passes through a 20 micron (absolute) bed filter to remove particles that may have passed through the depth filter.

After passing through the second filter, the NaK is collected in the precleaned barrel. When the barrel is full, as determined by weight change, approximately 2-5 psi argon cover gas is added and the barrel sealed.

B. DECONTAMINATION OF MERCURY PRESSURE TRANSDUCERS

Transducers previously used in Hg systems can be satisfactorily reused. However, when these are rewelded into the system, mercury that may be present can cause porosity in the weld. A procedure (Ref. 3), successfully used to remove all Hg prior to reinstallation, consists basically of subjecting the transducer to a 62% nitric acid (with 1 wt% sodium nitrite) solution while under vacuum. The acid removes any residual Hg not vaporized by the vacuum through a solution reaction.

C. SPECIFICATIONS

The following specifications and standards were published.

<u>No.</u>	<u>Title</u>
AGC-10319A	Cleaning and Drying of SNAP-8 Components and Systems, Procedure for
AGC-10319/6A	Cleaning of SNAP-8 PCS Components Prior to Final Assembly, Procedure for
AGC-10319/7	Removal of NaK Oxides and Hydroxides from Transducers, Procedure for
AGC-10319/8	Cleaning the Tube-in-Tube Boiler (Hg Side)

Report No. 3232

<u>No.</u>	<u>Title</u>
AGC-10319/9	Cleaning of Resin from Tube-in-Tube Boilers
AGC-10319/10A	Cleaning Mercury-Contaminated Components, Procedure for
AGC-10319/11	Cleaning Contaminated Transducers
AGC-10319/13	Cleaning, Condenser (Removing Mix-4P3E and Rubidium Oxide from the Condenser (Hg Side))
AGC-10319/14	Cleaning Oil-Contaminated Bourdon-Type Gages
AGC-10320B	Polyphenyl Ether "Mix-4P3E" Bis(Mix-Phenoxyphenyl)Ether
AGC-STD-1273A	Bending Tube & Pipe, 9Cr-1Mo, 300 Series, & 400 Series Steel

IV. TURBINE-ALTERNATOR ASSEMBLY

A. ALTERNATOR SEAL WELD EVALUATION

During PCS-1 Phase IV Step 2 operation, a loss in L/C vacuum was noted. Investigation revealed a defect in the alternator (S/N 481489), seal weld between the trunnion ring and the case adjacent to one of the trunnion bosses. The defect was repaired using mild steel weld filler metal and testing was resumed. After 143 hours of operation, a crack was found in the repair weld. The alternator was removed from the loop and was nondestructively evaluated to determine the failure mode. Based on the findings, it was subsequently deemed necessary to evaluate four other SNAP-8 alternators at Aerojet.

1. Evaluation of Alternator S/N 481489

The seal weld between the HY-80 trunnion ring and the mild steel case in the alternator was examined visually, and chemical analyses (Table 5) of filings from several weld areas and both parent metals were performed. No samples for metallographic analysis were taken (destructive evaluation procedure) because the unit had to be available for retest and so had to be repaired.

Examination of the original circumferential seal weld (disregarding the AGC repair weld at one trunnion boss) indicated the probability that no weld filler metal had been employed in the trunnion boss area when the unit was fabricated but that an iron base filler alloy had been used in the remainder of the weld. The drawing required the use of a ductile, non-hardenable, nickel-base alloy weld filler metal, Inco A. However, GE indicated that it was possible that no filler, 4130 alloy or mild steel filler, was employed. Chemical analysis of samples from the weld metal area which appeared to have filler added, and the weld metal area which appeared to be produced by fusion only, indicated neither was nickel base but rather both were of a hardenable iron-base composition. Qualitative hardness determinations indicated that the weld was harder than the parent metals and that the hardness increased in the area of the trunnion boss (Figure 5).

No preheat or postweld stress relief of the alternator seal weld was required on the drawing, and, according to GE, none was performed.

2. Postulated Failure Mechanism in Alternator S/N 481489

The chemistry of a weld deposit is controlled by the weld filler metal and by solution effects produced by melting of the parent metals on either side of the weld joint. With sufficient alloy pickup, a weld, even though deposited using a nonhardenable weld steel filler metal, may be rendered hardenable. The HY-80 alloy is hardenable, and a martensitic area in the weld-heat-affected zone would tend to form during the welding operation. The extent of this zone would depend on the weld-joint design and on the technique employed. This zone would tend to be less ductile than the parent

metal. The HY-80 would also contribute a sufficient amount of elements which enhance hardenability (chromium, molybdenum, and nickel) (Table 5) to produce a hardenable weld metal composition. The degree of hardening resulting in such a weld is, similar to HY-80, dependent on joint design and welding technique. Reduced ductility of such a weld varies directly as the increase in its hardness. A qualitative measure of this hardness increase is shown in Figure 5. The values are qualitative because of the measuring technique used to preserve the usability of the component. It is observed that, as the greater metal mass of the trunnion boss is approached, the weld metal hardness increases (conversely the ductility should be lower). This is because of the increased chilling effect on the deposited weld metal produced by the boss which acts as a heat sink. The smaller the distance between the weld and the boss, the faster the heat withdrawal. It is probable that a significant cause of the original failure of the weld at the trunnion boss was a low ductility weld resulting from the martensitic transformation of the weld metal and the HY-80 in the weld-heat-affected zone. Thermal and/or mechanical stresses imposed on the weld during system operation could produce a crack in the weld.

Although insufficient material to permit a complete analysis was obtainable from the Aerojet repair weld, it appears likely that the filler metal employed was a hardenable steel grade (MIL-R-5632 Type II), and alloying of the weld metal by adjacent molten material occurred. Hardening, and a resultant brittle structure, could have been produced in the same manner as in the original weld. This may have caused the service-induced crack which occurred in the repair weld.

3. Evaluation of Four Untested Alternators at Aerojet

Four other alternators were available for examination. These units are for future tests, and so the removal of material for chemical analysis was not performed. Only visual examination and chemical spot tests were made. A marked variation among the four alternators was found in the seal weld between the HY-80 trunnion ring and the mild steel case among the four (Table 6). There are variations between alternators in the amount and type of weld filler metal used, in the weld configuration and, therefore, presumably in the weld metal structure.

The S/N 481492 alternator has the same weld configuration at the trunnion boss as the S/N 481489 alternator (i.e., little or no added weld filler metal) and presumably would be subject to the same type of failure in service. The S/N 481490 alternator appears to contain an iron-base filler at the trunnion boss and also could be subject to failure at this point depending on the exact filler metal composition. If it is a hardenable alloy such as 4130, which appears possible based on information from GE, difficulty would be anticipated. The other two alternators, S/N 481510 and S/N 481491, have nickel-base weld filler metal at the trunnion bosses and would be less apt to fail at the boss. However, it appears that the nickel-base filler may not have been deposited over an area sufficient to minimize the potential for failure. Hardness measurements indicate this deposit should extend at least one or two inches beyond each end of each boss where significant hardening due to boss proximity occurs. Such an extension of the deposit was not found.

All four of these alternators, however, have either no filler metal or have an iron-base filler metal in the areas away from the trunnion. Therefore, it appears possible that these joints could be brittle and that failure by cracking in service could occur if service stresses, either transient or steady-state are large enough.

4. Discussion

HY-80 in the quenched and tempered condition is a low alloy, high-strength steel (typical of a group of constructional steels) employed frequently for the hulls of warships and submarines. In these applications, welding is performed; however, multiple passes employing filler metal are used so that subsequent weld passes temper the previous weld metal and the heat-affected zone of the parent metal. For small structures, stress relieving in a furnace is often employed to temper the joints. However, postweld heat treatment is not mandatory. As in many welding applications, the requirement depends on joint design and other welding variables. Mild steel can be welded without producing a brittle joint because the alloy content is so low that extremely rapid cooling rates, such as brine quenching, are necessary to produce martensite.

A fusion weld of mild steel to HY-80 without the use of filler metal produces a weld metal deposit which falls in the range of the 4300 alloy series, an iron-base alloy with chromium, nickel and molybdenum. This alloy is hardenable. The degree of hardening obtainable depends on the carbon content and on the cooling rate from the austenitizing temperature. The relatively massive, unpreheated, HY-80 section caused rapid cooling of the weld metal and the heat-affected zone. In general, the higher the carbon content and the higher the cooling rate, the higher the hardness obtained and consequently the less ductile the material. The brittleness of this structure may be reduced by an elevated temperature stress relief or tempering operation.

Employing Inco A as a filler metal would eliminate the brittleness in the weld metal itself because this is a ductile non-hardenable alloy. There would, however, tend to be a hardened area in the HY-80 heat-affected zone of the parent metal. The extent of this hardening would depend on the joint design and the welding technique. The extent of hardening in the seal welds of the alternators evaluated cannot be predicted from the examination performed. Destructive metallographic examination and hardness measurements are required. If subsequent passes had been made over the entire seal weld and the temperature of the HY-80 in the heat-affected zone had not exceeded approximately 1300°F, improved ductility in the weld would have resulted due to tempering of the martensitic structure. As an alternative, the optimum weld metal and heat-affected zone structure could be produced by stress relieving the joint. Since stress relieving of the alternator joints is not considered practical, the acceptability as a compromise of the multiple weld pass method for producing the joint with Inco A filler should be evaluated. A preheat and interpass temperature of 200°F to 250°F should be employed. Also the stresses imposed on this joint should be determined to assure they are not higher than permissible.

5. Conclusions and Recommendations

The conclusions resulting from this investigation were:

- a. A low ductility weldment acted upon by stresses imposed on the joint in service was a prime contributor to the seal weld failure in the S/N 481489 alternator. The weld metal deposit was a hardenable composition which was quenched from the welding temperature by the massive, unpreheated HY-80 section.
- b. The other four alternators at Aerojet are subject in varying degrees, to seal weld cracking due to relatively low ductility weldments.
- c. The alternator drawing requirement for the use of Inco A filler metal to produce the HY-80 to mild steel seal weld was not met except for localized areas on two alternators.

Two recommendations were made to avoid future failures. First, the entire seal weld between the HY-80 trunnion ring and the mild steel case in all alternators at Aerojet should be repaired by using multiple passes with Inco A filler wire and a preheat and interpass temperature of 200°F to 250°F. This repair should be simulated with test joints to demonstrate its adequacy. Second, for future units, the seal-weld design, materials selection, and fabrication processing should be reevaluated to eliminate the possibility of producing brittle welds which are subject to failure in service.

B. TURBINE

1. Turbine Industry Survey Trip

During the period of 29-31 March 1966, the following companies were visited to discuss SNAP-8 turbine materials:

Haynes Stellite, Kokomo, Ind.
Allison Division, General Motors, Indianapolis, Ind.
General Electric, Evandale, Ohio
Westinghouse Astronuclear Laboratory, Large, Pa.

Opinions were solicited on the following general subjects:

- a. Stabilization of FCC Stellite 6B under SNAP-8 operating conditions,
- b. General material requirements specified for use in their respective turbines,
- c. Specific materials used and turbine operating, or test history relative to each,

- d. The desirability of SNAP-8 use of HCP Stellite 6B, and
- e. Other sources that might have additional data on turbine materials experience.

The following is a summarization of the information gathered during the several visits.

The metallurgy of Stellite 6B has not been studied intensively. No more than conjecture on stabilization of FCC appears possible with the currently available technology. The FCC material appears to be thermodynamically unstable. However, the time-temperature-structure relationship is unknown except for SNAP-8 work.

The opinions of aircraft turbine manufacturers support the use of ductile blade materials. The quoted minimum lies between 5 and 10% elongation at room temperature; however, history of the use of less ductile materials exists.

There is little experience with integrally cast wheels. Mechanical or fir-tree attachment is generally used.

New alloys are introduced reluctantly and only after extensive testing. The turbine industry would rather spend more effort on cooling procedures to gain the last bit of capability from established alloys.

2. Stellite 6B (HCP) Mechanical Properties

To establish the suitability of Stellite 6B in the hardened and HCP condition for use in the SNAP-8 turbine assembly, a program was initiated to statistically establish the mechanical properties of the material. Room-temperature and elevated-temperature tensile and Charpy V-notch impact tests will be performed. The results of these tests can then be correlated with the properties of Stellite 6B in the FCC condition and with the properties of other materials employed in gas-turbine applications to permit the evaluation of Stellite 6B as the SNAP-8 aerodynamic material.

a. Transformation of Specimen Material

Stellite 6B bar stock was procured to determine its properties. The rough machining of impact and tensile specimens was initiated and, concurrently, samples of the bar stock were thermally exposed to determine an optimized cycle for crystal structure transformation to HCP, and for hardening. This lot of material did not respond to the previously developed transformation-hardening cycle of 1650°F for 4 hours plus 1250°F for 48 hours. The table below summarizes the results of the thermal transformation treatment.

Thermal Transformation of Stellite 6B
(1650°F/4 Hour Followed by 1250°F/48 Hour)

	HCP Content <u>%</u>	Hardness <u>R_c</u>
Pretreated material	6.7	37
Desired condition	80 min	48.5 min
Post-treated material	30	39

Groups of samples were exposed at 1250°F, 1350°F, 1450°F, and 1550°F after an initial treatment of 1650°F for 4 hours. Results (Ref. 4) indicated that the transformation occurred most rapidly with the 1450°F exposure. Therefore, exposure of the tensile and impact test specimens was initiated at 1450°F.

The results of X-ray diffraction analyses and hardness determinations indicate that a considerable variation exists in the transformation and hardening rates of this heat of 5/8-in.-dia bar stock, and that the reaction is extremely sluggish. Nineteen specimens, after 570 hours of exposure at 1450°F, exhibited variations between 80 and 92% HCP and a hardness range from R_C 45 to R_C 50. Another group of 25 specimens, after 235 hours of exposure, exhibited a variation between 54 and 85% HCP and a hardness range from R_C 44 to R_C 49.

b. Status

Previous data obtained by Aerojet and by the Cobalt Information Center indicate that the specimens can be considered transformed to HCP when their hardness is R_C 48.5, minimum, and the amount of HCP is 80%, minimum. Based on this criterion, all impact specimens were transformed as a result of exposure times varying from 235 to 1100 hours at 1450°F. Final machining of specimens is in process. None of the tensile specimens have reached the accepted condition after times up to 800 hours at 1450°F. Transformation and hardening are continuing slowly.

3. Turbine Material Metallurgical Stability

a. Stellite 6B (FCC)

A section of the turbine assembly TA 3/2 Stellite 6B diaphragm was exposed at 800°F for 1025 hours after being rapidly quenched to 1000°F in a salt bath from the 2250°F annealing temperature. The FCC structure of the material was stable throughout the exposure period at 800°F. The specimen was then inserted in a 1200°F furnace for an aging test. After 1710 hours at this temperature, the hardness of the specimen had increased to R_C 56 from the pre-exposure hardness of R_C 40 and the structure had transformed to all HCP from the FCC structure as indicated by diffractometer X-ray diffraction analysis. It therefore appears extremely unlikely that the FCC structure can be stabilized at the 1200°F turbine operating temperature.

The salt-quenched section of the fourth-stage Stellite 6B diaphragm being exposed at 1065°F showed no further change after 3955 hours either in hardness or amount of HCP present after the initial minor changes during the initial 400 hours of exposure. It appears that the FCC structure of Stellite 6B can be stabilized for service temperatures below 1065°F.

b. S-816 Stability

After 4275 hours of exposure at 1200°F, the hardness and crystal structure of a S-816 specimen did not change from the original heat-treated condition. The hardness remained R_C 30 and only the FCC phase was detectable by X-ray diffraction analysis. The metallographic structure indicated the possibility that a small amount of sigma phase may have formed during the exposure (Figure 6). However the amount and distribution appears to be such, if in fact it is sigma, that no serious effect on the properties of the material would be anticipated.

4. PH 15-7 Mo Second-Stage Shroud

Redesign of the SNAP-8 turbine assembly second-stage nozzle diaphragm, during the latter part of 1965, incorporated the use of a dissimilar metal shroud mechanically attached to the Stellite 6B diaphragm. A design requirement was that the coefficient of expansion of the shroud material should be no greater than that of Stellite 6B. Of the candidate alloys investigated, PH 15-7 Mo (heat treated to TH 1050 condition) was selected for second-stage shroud use because it appeared to meet the design requirements and it was most readily available.

Distortion of the PH 15-7 Mo second-stage shrouds removed from the TA 2/3 and TA 5/2 turbine assemblies was noted after 143 and 42 hours of operation respectively. The parts were axially warped and also exhibited ovality. The excessive distortion of the shrouds was unacceptable.

Therefore, an investigation (Ref. 5) of the precipitation hardenable stainless steel, PH 15-7 Mo, was conducted to determine if metallurgical instability had contributed to the distortion, in service, of the SNAP-8 turbine second-stage shrouds fabricated from this alloy. It was determined that this material in the TH 1050 condition is metallurgically unstable and that dimensional changes would occur at the service temperature, 1065°F. An in-fabrication thermal treatment was developed (a 50-hour soak at 1065°F after the TH 1050 heat treatment) to produce stability prior to final machining of components. It was also determined that considerable variation in response to this treatment between lots should be anticipated. It was recommended that PH 15-7 Mo be employed only as an interim alloy for SNAP-8 turbine assembly applications and that components of an alternative alloy, S-816 or Lapelloy, should be substituted. Estimates of the mechanical and physical properties of the specific lots of material investigated, in the stabilized condition, were made.

5. Inconel 718 Retainer Spring

Metallographic examination of the Inconel 718 retainer spring from the first-stage of TA 6/1 after 117 hours of operation indicated that the iron and nickel plating had not been affected. The iron plating was intact and was uniformly 0.93 mils thick; the electroless nickel plating over the iron to prevent atmospheric corrosion was uniformly 0.13 mils thick and showed no evidence of mercury corrosion or loss of integrity. These dimensions are essentially identical with those obtained from another retainer in the as-plated condition. The hardness of the base metal had not altered from the original R_C 44 by the operational exposure. It was concluded that the Inconel 718 is adequate for the intended application from a metallurgical standpoint and that possibly alloys with relatively high nickel contents may be usable in the dryer stages of the turbine without catastrophic Hg corrosion occurring.

6. Interstage Pressure Tap Joint

The turbine assembly interstage pressure taps consist of mild steel pipe threaded into the 9Cr-1Mo turbine case employing a Dryseal

pipe thread. The Dryseal thread provides the Hg seal and an Easy Flo silver brazing alloy joint prevents loosening of the threaded joint by operational vibrations and, coincidentally, provides a backup seal. This joint design was selected as the most practical to incorporate in finish-machined turbine assembly housings (the only type available). The maximum service temperature of these joints is 1000°F. The joint configuration is illustrated in Figure 7a. Figure 7a also illustrates the mechanical seal provided by the threads and the labyrinth passage formed from the inside of the turbine assembly to the exterior braze joint, a passage which should minimize, if not prevent, liquid Hg exposure of the braze during turbine assembly operation.

a. Elevated Temperature Integrity of Braze Joint

Typical sections representing the optimum braze joint configuration which was incorporated in TA 5-2 (currently being installed for PCS-1 Phase IV for Step 2 testing) were exposed at 1000°F in air for 1968 hours to determine if the operating environment will affect the integrity of the joint. Metallographic examination of individual specimens after various periods of exposure (Figure 7b) indicated that the joint had retained its integrity without identifiable diffusion effects. It was concluded that this joint configuration would be adequate for a metallurgical standpoint for periods of at least 2000 hours and potentially for considerably longer periods of time. It appeared that oxidation of the mild steel would be the limiting factor in the life of the joint.

b. Braze Joint Properties in TA S/N 2-3 Unit

It might be desirable in the future to break these joints to remove the taps, or an unsatisfactory design may result in excessive braze joint leakage during Hg loop prestart evaluation. These appear as potential problems because of two unfavorable conditions. First, the in-situ brazing of the leaking pressure taps on TA 2/3 in PCS-1 during the Phase IV Step 2 was performed under adverse circumstances of cleanliness and accessibility. Second, 316 SS pressure tap fittings were employed in this particular TA assembly. Because of the higher coefficient of expansion of 316 SS compared to the 9Cr-1Mo steel housing, a high tensile stress on the braze material could occur during cooldown after system operation. Lap shear type tests were performed to determine the approximate remelt temperature of a wide gap joint and the lap-shear strength of the joint near the melting temperature. This information would aid in tap removal for rebrazing should it prove necessary.

The brazed specimens were suspended vertically with a 9.5-lb weight suspended from the lower end. Thermocouples were capacitance discharge welded to the steel at the joint. The specimens were heated by induction to 1000°F and stabilized at that temperature for approximately 15 seconds. The temperature was then increased at an average rate of 75°F/min until specimen separation occurred. All of the specimens failed in the braze alloy at an average temperature of 1145°F and an average lap-shear strength of 47 psi. It appeared feasible to remove the taps from the turbine assembly if necessary by heating combined with mechanical unthreading without exceeding the transformation temperature of the 9Cr-1Mo steel. Apparently because of the test specimen joint's wide gap,

the remelt temperature of the braze alloy is identical to the original melting temperature. With controlled gap brazing (0.002 to 0.004 in.) the remelt temperature would tend to be higher due to alloy effects with the base metal (Ref. 6). Such a design exists in the TA 5/2 unit. Therefore, these tests will be duplicated with smaller gaps to provide necessary information for rework preplanning.

V. EVALUATION OF -1 BOILER (P/N 092020-1F, S/N A-1)

The interface component between the NaK primary loop and the Hg loop in the SNAP-8 system is the boiler. This component is used to transfer heat from the NaK to the Hg, producing Hg vapor to drive the turbine-alternator assembly. The -1 boiler (Figure 8) is a combination cross-counter flow, tube-in-shell heat exchanger. The mercury flow in four 60-ft-long tubes coiled on two double-lead helices. A plug to restrict Hg flow is placed in the Hg inlet end of each of the four parallel flow passages. The plug in this restricted flow section is a solid rod centered within the tube and separated from the tube wall by a 0.135-in. diameter wire wrapped around the rod. The wire forms a spiral flow path for the mercury. This plug insert continues through the boiler for 10 ft. Downstream of the plug, the spiral Hg flow is maintained by a twisted ribbon insert with a pitch of 7.2 in. which continues for the remainder of the boiler length. The swirl flow serves to separate the high-density liquid from the vapor, making the boiler operation insensitive to gravity and increasing heat-transfer rates. The mercury coils are surrounded by two concentric cylindrical shells which form an annular flow passage for the reactor coolant, NaK-78 (the eutectic mixture of sodium and potassium). The Hg tubes are 0.902-in. ID by 0.125-in. wall 9Cr-1Mo steel, and the shell is 316 SS. The plug consists of a 0.600-in. OD low-carbon steel rod and low-carbon steel wire. The ribbon is also of low-carbon steel, 0.016-in. thick.

Loop testing of the boiler (S/N A-1) was initiated in July 1964 using the RPL-2 facility. After successful completion of a performance test program in July 1965, the boiler was removed from the loop and a metallurgical evaluation was conducted.

A. OPERATING HISTORY1. NaK Side

The nominal rated design conditions on the NaK side of the boiler are listed in Table 7. At the time of installation of the -1 boiler in the RPL-2, the NaK primary loop was operating without a NaK purification system. Design and operating difficulties were experienced intermittently. Purification, to whatever level was achieved, was accomplished during the initial period of boiler operation - primarily through multiple hot dumps prior to each operating period. The resultant oxygen contamination was sufficient to produce significant mass transfer during this oxygen-contaminated operating period in many of the components, the EM pump, and the venturi flow meters. Toward the middle of the test period, the cold trap of the purification system became operative. Although plugging meter runs were not made, it is estimated, based on periodic examination of components (the absence of NaK mass-transfer deposits), and the cold trap operating temperature 200-250°F, that an oxygen content of 25-40 ppm was maintained during the last 1140 hours of a total NaK-side operating time of 2350 hours.

2. Hg Side

The rated design parameters of the Hg side of the boiler, and typical performance characteristics during the test period, are shown in Table 7. The boiler started the test series with a less-than-satisfactory heat-transfer capability (unconditioned state), and saturated vapor was not produced (Ref. 7). From November 1964 through March 1965, the boiler was operated intermittently for a total of approximately 300 hr with a rubidium (Rb) additive in the Hg to promote full conditioning and to attain rated boiler outlet conditions. Testing continued thereafter without Rb until a total of 1415 operating hours were logged. During the last half of the test period (approximately 700 hr), boiler characterization tests were conducted under varying off-rated operating conditions.

B. Hg INLET PLUG

The mild steel bar and mild steel wire that comprised the Hg inlet plug (Figure 9), contained visual surface defects resulting from boiler operation. The more severe attack, Figure 9 and Table 8, was found on the wire in the tight-pitch (high velocity) length. All estimates of defect depth are based on the assumption that high points in the micrographs represent the pretest metal surface position.

The wire was wound around the plug such that there existed a gap between the two. This gap apparently allowed bypass of the Hg resulting in attack of the wire surface at the interface at a rate of 3 mils/1000 hours. Although there is no evidence that the attack of the plug caused reduced boiler performance in the total 1425 hours of Hg-side operation, it is presumed that continued operation would have ultimately degraded the heat transfer. The degradation would have been caused by a decreased liquid-Hg velocity through the plug area. The Hg velocity decreases when wire material is removed and Hg flow assumes more of an axial flow direction rather than spiral flow through the helical channel formed by an unaffected wire. This heat-transfer degradation by wire attack probably would be counterbalanced somewhat by increased surface wetting of the plug length surfaces by the Hg with continued boiler operation. Wetted surfaces add materially in effecting optimum heat transfer to the boiler. There are no previous test data on which to base an estimate of the time required to produce measurable degradation.

C. TWISTED RIBBON TURBULATOR

The twisted ribbon turbulator located downstream of the plug contained areas of mass-transfer material deposition which visually appeared uniform but which was, in fact, spongy and dendritic and existed as dispersed individual accumulations (Figure 10). Along the length of the tube from the Hg inlet, the height of the deposit increased to a maximum of 3.3 mils at approximately 46 ft, 8 in. from the inlet.

X-ray examination of tubes (Ref. 1) showed that the ribbon contained areas of local distortion which existed during boiler operation. These areas

apparently acted as corrosion product traps where unusually large accumulations of material were found, 15 mils thick or greater, causing an unknown amount of reduction in the tendency for product carryover to the turbine.

D. EXTRAPOLATED BOILER TUBE LIFE

Three 9Cr-1Mo steel boilers have been operated and evaluated as part of the SNAP-8 program. Two were 1/19 scale models and the third was the -1 full-scale configuration. The test parameters varied sufficiently between these three that it is impractical to attempt a correlation of the data. Qualitatively, these tests indicated that 9Cr-1Mo steel may be marginal for 10,000-hour service. At the same time, the observations made during these tests should be of significant value in the ultimate reliable estimation of 9Cr-1Mo steel boiler life. These observations were:

- The liquid Hg velocity has a distinct effect on boiler conditioning.
- The attack pattern in the boiler depends on the conditioned state during boiler operation.
- The maximum attack is apparently associated with the liquid-Hg section and the high-heat-transfer area of the boiler tube.

The tests are described below and the resultant data are used to estimate the available maximum life of each boiler utilizing an assumption of an increased wall thickness to .200 in. (assuming that this thickness would satisfactorily transmit heat). The extremely conservative assumption is made that penetration through the added wall thickness can be accepted and that maximum attainable life is defined by the period required to penetrate to the point where the original 0.090-in. wall remains.

1. CL-3 Subscale Boiler Test

Figure 11 describes a 1/19 scale tube-in-tube boiler test in the Corrosion Loop 3 (CL-3) facility. The Hg temperature profile was deduced from actual NaK temperature measurements. The pit depth curve resulted from measurements of the maximum depth made at various points along the length of the tube. At the start of this test, the boiler operated in a deconditioned state. During operation the performance of the boiler continued to improve. The boiler became more and more conditioned and the high-heat-transfer area, as defined by the steep slope portion of the Hg temperature profile curve, continuously shifted from the Hg outlet end to the left and ultimately reached the position shown. The curve represents steady-state conditioned operation for the last 2200 hours of the 4400-hour test. It appears that the pattern of penetration distribution along the tube length is associated with: (a) the variable operating condition in that the penetration appears to have occurred along a significant length of the tube, and (b) the moving high-heat-transfer area, where the maximum pit depth, for example, appears associated with the location of the high-heat-transfer area during the longest portion of the test.

With the conservative assumptions described in the paragraph immediately above, and using the maximum pit depth and the assumed associated 2200 hours required to develop this pit, it is estimated that a boiler of this type with 0.2-in. thick tube walls would have operated between 5000 and 6000 hours prior to failure.

2. CL-4 Subscale Boiler Test

Figure 12 illustrates a second 1/19 subscale boiler test conducted in Corrosion Loop 4. CL-4 was of identical design to CL-3. It also tested a tube-in-tube boiler of the same design except that the Hg inlet plug was changed after an initial deconditioned operating period to significantly increase the liquid Hg velocity from 0.9 to 6 fps. This velocity change produced an immediate effect on boiler performance. During the initial 650-hour operating period, this boiler exhibited a mercury temperature profile with the high-heat-transfer area located approximately at the Hg outlet similar to the CL-3 boiler. The increased velocity immediately conditioned the boiler altering the Hg temperature profile curve in one step where the high-heat-transfer area suddenly shifted to the point indicated in Figure 12. Comparison of this figure with Figure 11, indicates that this high liquid-Hg velocity change produced a marked difference in the tube wall penetration pattern of CL-4 compared to CL-3. Two distinct areas of penetration are observed in CL-4. The first at the boiler outlet which appears associated with the initial 650 hours of deconditioned operation and another area at the boiler tube inlet which appears associated with the high-heat-transfer area of the mercury temperature profile which existed during the final 1850 hours of steady-state conditioned operation. Making the same assumptions as with CL-3, a calculation was made that predicted a maximum attainable life a boiler of this type with 0.2-in. thick walls of approximately 7000 hours.

3. -1 Boiler Estimated Life

Figure 13 illustrates a test of the full-scale tube-in-shell -1 boiler (P/N 092020-1F S/N A-1) conducted in the RPL-2 facility. The varying conditioned state of this boiler was comparable to that experienced in the CL-3 test. That is, the boiler performed in a deconditioned state initially and, as operation continued, performance improved and the high-heat-transfer area of the Hg temperature profile moved from an initial location at the boiler outlet to the left until it ultimately reached the position represented by curve B. The penetration distribution pattern along the length of the tube also appears to have been generated by the same phenomena as in CL-3; i.e., the continually improved conditioning with operation and the continuous movement of the high-heat-transfer area of the Hg temperature profile curve. Thus, the results appear to confirm the assumption of the qualitative correlation of the penetration pattern with continued boiler conditioning. By making the same assumptions as were made for the previous two subscale boiler tests, it is calculated that the maximum attainable life for a boiler of this type with 0.2-in. thick walls was approximately 11,000 hours. Thus, the corrosion rate does not appear excessive for SNAP-8 life requirements.

These three boiler tests were run under such diverse conditions that correlation of the data to arrive at a reliable conclusion as to the capability of 9Cr-1Mo steel to meet SNAP-8 life requirements is not feasible. However, these data plus those of other tests both completed and in process will result in an analytical model which relates Hg corrosion potential to boiler design and operating variables. This model will indicate the parameters, and the control required to reduce the corrosion potential of the SNAP-8 boiler material. With this information, a 9Cr-1Mo boiler which exhibits minimum Hg corrosion potential will be designed, fabricated and tested to confirm the anticipated life of 9Cr-1Mo steel. The test is scheduled to start in the Fall of 1966.

VI. CORROSION LOOP PROGRAM

The objectives of this program are to determine corrosion and mass-transfer patterns in the mercury and NaK loops of the SNAP-8 system, and to evaluate the corrosion resistance of the SNAP-8 reference materials with regard to the 10,000-hour life requirement. In addition, the corrosion loops have been used to investigate mercury boiler heat-transfer characteristics.

Corrosion loops 1 and 2 (CL-1 and CL-2) were constructed of Haynes 25 alloy. Operation of the first loop was completed in 1962. The second loop was converted into component test loop 2 (CTL-2) that was operated to check the performance of certain components to be used in subsequent loops and to run mercury boiler performance tests. Operation of CTL-2 was completed in 1965.

The mercury containment material for CL-3 and -4 was 9Cr-1Mo steel. The NaK primary loop was constructed of 316 SS with a section of Hastelloy C in the low-temperature area and sections of chromized Hastelloy N and 347 SS in the high-temperature area. Type 316 SS was used for the NaK condensing loop.

Operation of CL-3 was completed in December 1964 and was disassembled for evaluation. Corrosion loop 4 was started in January 1965 and the operation of the loop through December 1965 was mainly to test mercury boiler performance, as reported in Reference 1.

In January 1966, the emphasis of the Corrosion Loop Program was shifted from boiler performance testing to a program with the objective of determining the maximum potential life of a SNAP-8-type mercury boiler using 9Cr-1Mo steel as the mercury containment material.

The approach to the new objective is outlined as follows:

- Set up a mathematical model that describes the corrosion in the preheat section of a SNAP-8-type mercury boiler and conduct a parametric analysis of the boiler to determine the design parameters that can be modified to lower the mercury corrosion rate in the boiler.
- Conduct capsule tests to develop a procedure that will assure complete local wetting of the mercury boiler containment tube. Complete mercury wetting was considered necessary so that reproducible results could be achieved in loop tests designed to test the model that describes the corrosion in the boiler.
- Modify CL-4 boiler so that short-term preliminary tests can be made to test the wetting procedure and the results of the parametric analysis that describes the corrosion in the boiler.
- Using the results of the short-term tests, design and operate a mercury boiler in CL-4 for an extended period (~2500 hours) to determine the corrosion pattern and rate in a 9Cr-1Mo boiler.

In addition to the work planned with CL-4, the planning for a single-phase liquid mercury loop designated corrosion mechanism loop 1 (CML-1) was initiated. The objective of this loop is to determine the mechanism that controls the corrosion of 9Cr-1Mo in mercury.

A. CORROSION LOOP 4 BOILER EVALUATION

Corrosion Loop 4 was shut down after the completion of the gravity-type separator tests (Ref. 1) and the mercury boiler was removed for evaluation. The CL-4 mercury boiler is described in Reference 8. The total time of mercury boiling was 2576 hours which included the following tests:

650-hour corrosion test

564 hours for heat-transfer tests

830 hours for mix-4P3E injection and conditioning tests

359-hour multimetal plug corrosion test

173-hour mix-4P3E separator test

Various NaK profiles were generated during these tests; however, most of these profiles can be represented by two typical profiles. The characteristic profile for the 650-hour corrosion run appears as Curve 1 on Figure 14. Mercury boiling for this profile occurred 50 to 60 ft from the mercury inlet. The other profile (Figure 14, Curve 2) is the characteristic profile for the last 1926 hours of mercury boiling. For this profile mercury boiling occurred 2 to 8 ft from the mercury inlet.

After the boiler was removed from the loop, the tubing was split longitudinally using a band saw.

1. NaK Side of the Boiler

a. 316 Stainless Steel Tubing

The 316 SS outer shell of the mercury boiler visually showed no evidence of corrosion or mass transfer.

b. Outside Diameter of the 9Cr-1Mo Tubing

Pitting and metallurgical change were evident at the OD of the 9Cr-1Mo boiler tubing (Figure 15). Pits, 0.001 to 0.0015 in. deep, were present along the entire length of the boiler section. Decarburization and grain growth began about 15 to 20 ft from the mercury boiler inlet. There was a gradual increase in the amount of decarburization and grain growth as the distance from the Hg inlet increased and, consequently, as the NaK temperature increased. Prior analysis (CL-3 boiler) indicated that this type of decarburization and grain growth are typical for 9Cr-1Mo steel in a SNAP-8-type boiler.

2. Mercury Side of 9Cr-1Mo Tubing

a. Coil Section

One area of pronounced pitting occurred in the coiled section of the mercury boiler. This area was approximately 50 to 60 ft from the mercury inlet. The location of this pitting area coincides with the maximum heat flux area for the 650-hr corrosion run (Figure 14). The deepest pit in this area was 0.0065 in. The pits assumed one of three different configurations: isolated single pits, pits combined into straight line segments, and pits combined in the form of circular dished out areas. The link segments were about 1-1/2 in. apart, with the circular dished out areas between them.

Corrosion product deposition (Figure 16) was found immediately after the boiler inlet plug region (5 to 12 ft from the mercury inlet) with a maximum thickness of 0.004 in. There were minor deposits at the end of the boiler (50 ft from the mercury inlet) that were approximately 0.001 in. thick.

Examination of the microstructure of the inside diameter of the tubing indicated a white layer approximately 0.0001 in. thick along the coiled section of the tubing. Other than this layer, there were no other changes in the microstructure of the 9Cr-1Mo.

b. Boiler Inlet Plug Section

In the CL-4 boiler, the area of major pitting was 17 to 26 in. from the mercury inlet or in the preheat section of the boiler (Figure 17). In this area a large percentage of the pits had united to form transverse grooves (0.0265 in. deep max) and shallower grooves at 20° from the transverse position. The groove configuration indicates the effect of the tight pitch region of the various multipitch plugs operated in this boiler. The probability that some form of tube wall corrosion would appear in this area was high since corrosion was noted in the tight-pitch region of the multipitch plugs when they were removed after the boiler performance tests.

Examination of the microstructures of the tubing in this section showed no white layer on the surface. The microstructures are typical of 9Cr-1Mo exposed to general solution attack by mercury.

3. Discussion

The CL-4 boiler was used for a variety of corrosion tests and boiler performance tests. During the boiler performance tests, the boiler inlet plug was changed frequently so the corrosion pattern in the boiler cannot be related to the operating time with a specific boiler inlet plug.

In general, the pitting at the end of the mercury tubing (50 ft from the mercury inlet) in the boiler is considered to be associated with the first 650 hours of operation when the boiler was deconditioned and the mercury boiling was taking place at the end of the boiler. The deep grooves in the boiler

inlet plug section 17 to 26 in. from the mercury inlet are considered to be the result of the last 1926 hours of operation when multipitch boiler inlet plugs were used. This boiler does illustrate the influence of heat-transfer performance on the corrosion pattern of a SNAP-8-type mercury boiler constructed of 9Cr-1Mo.

No ID or OD cracking of the 9Cr-1Mo mercury containment tubing in the entire boiler length was observed as was seen when CL-3 boiler tubing was evaluated (Ref. 9).

B. PARAMETRIC ANALYSIS OF MERCURY CORROSION

An analytical program was initiated during this report period to determine the SNAP-8 tube-in-tube boiler design parameters that could be modified to minimize the corrosion potential of 9Cr-1Mo steel. To achieve the above, the program was to develop the following:

A corrosion model based on mass-transfer consideration.

An analytical technique utilizing the above model.

A computer code to facilitate the calculation.

The manner in which the program was conducted is as follows:

1. The basic SNAP-8 tube-in-tube boiler design was examined to determine which design parameters could be easily modified.
2. Each of these parameters were varied one at a time over a small range to determine which one yielded the most significant reduction of corrosion.
3. Combinations of the above parameters were varied over a large but reasonable range to determine their effect on corrosion performance.
4. Examine the results for significant trend.

The parameters selected to be varied are as follows:

NaK jacket ID

Mercury tube ID and for the plug insert

Thread height

Thread pitch

Thread width

The number of mercury passages per tube

To provide a basis for comparison, the present SNAP-8 tube-in-tube boiler, as described in Reference 10, was run through the computer code to determine its corrosion behavior. A summary of the results may be found in Figure 18. The parameters listed above were then varied $\pm 5\%$ to determine the effect on the corrosion behavior. The results are summarized in Tables 9 and 10. Examination

of these results indicates that the thread pitch, thread height, and number of mercury flow channels per tube yield the most significant change in the mercury corrosion behavior. Further examination of the effect of the number of mercury channels per tube shows that, although the maximum wall penetration is decreasing, the total corrosion as indicated by the exit concentration of iron is increasing. This is due to the longer preheat length required as the number of channels is increased, thereby distributing the corrosion over a larger area. Also associated with this increase in preheat length is an increase in the Hg side ΔP in the preheat region. Therefore, the only parameters that were considered further are thread pitch and thread height. A parametric study was set up for the computer code to vary the thread pitch from 0.375 to 2.25 in. and the thread height from 0.062 to 0.122 in. The results are shown in Figures 19 and 20 and in Table 11. It was at this point that the most significant conclusion was reached: in order to decrease the corrosion of 9Cr-1Mo by Hg, the mass velocity (or its equivalent, the mercury liquid-phase velocity) must be decreased. Associated with this decrease in mass velocity is the decrease in the liquid-phase pressure drop.

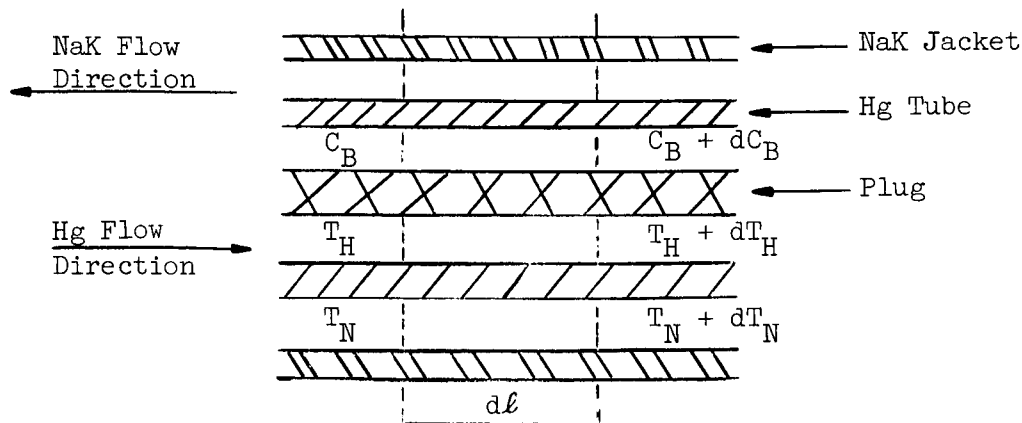
The following is a discussion of the computer adaptation of the analytical technique and the results of the analysis.

1. Analytical Technique Adaptation - Computer Code SECAT

SECAT (SNAP Eight Corrosion And Thermal Analysis Code) is an IBM-7094 computer code written in FORTRAN and formulated to predict the corrosion and thermal performance of the preheat region of the SNAP-8 tube-in-tube boiler. For a given set of operating conditions and NaK and mercury flow channel geometries, SECAT will calculate the required heat-transfer length and corrosion performance of the preheat region of the boiler. The method employed by the code is numerically integrated by a Runge Kutta scheme, the differential equations defining the spatial behavior of the heat transfer and corrosion processes. In addition, the code will calculate the NaK-side and Hg-side pressure drops. The code output is a tabulation of the calculated axial variation of the significant performance parameters in the preheat region.

The differential equations used in SECAT are derived as follows:

Consider a differential length of the preheat region as shown in the following diagram. Although an annular geometry is shown for both the NaK and Hg side, any geometry may be analyzed by using the appropriate geometric factors and fluid-flow and heat-transfer correlations.



The rate of heat transfer between the two streams may be expressed by the following three equations:

$$q = W_N C_{PN} dT_N \quad (1)$$

$$q = W_H C_{PH} dT_H \quad (2)$$

$$q = U_O P_{mt} (T_N - T_H) d\ell \quad (3)$$

where

q = rate of heat transfer

W = flow rate

C_P = heat capacity

U_O = overall heat transfer coefficient

T = temperature

P = perimeter

ℓ = length or position parameter

Subscripts

N refers to NaK

H refers to Hg

mt refers to Hg tube

The mass flux of Fe from the surfaces in contact with Hg (Hg tube and the plug) may be expressed as follows:

$$N_{mt} = K_{O_H} (C_{mt}^S - C_B) \quad (4)^*$$

$$N_{Pl} = K_{O_H} (C_{Pl}^S - C_B) \quad (5)^*$$

where

N = mass flux of iron

K = mass-transfer coefficient

ρ = density

C = Fe concentration in Hg

* It must be noted that these equations are for 9Cr-1Mo steel operating under fully wetted conditions in addition to assuming that the boundary layer diffusion model predicts the corrosion behavior.

Subscripts/Superscripts

P_l refers to plug

s refers to saturated value

The terms C_{mt}^s and C_{pl}^s are the saturation concentrations of Fe in Hg evaluated at the tube and plug surface temperatures, respectively. Knowing the mass flux, one can easily write a mass balance equation for the Fe concentration.

$$dC_B = \frac{K\rho_H}{W_H} \left[P_{mt} (C_{mt}^s - C_B) + P_{pl} (C_{pl}^s - C_B) \right] dl \quad (6)$$

Algebraic manipulation of the heat and mass balance equations yield the following:

$$\frac{dT_H}{dl} = \frac{U_o P}{W_H C_{PH}} (T_N - T_H) \quad (7)$$

$$\frac{dT_N}{dl} = \frac{U_o P}{W_H C_{PN}} (T_N - T_H) \quad (8)$$

$$\frac{dC_B}{dl} = \frac{K\rho_H}{W_H} \left\{ P_{mt} (C_{mt}^s - C_B) + P_{pl} (C_{pl}^s - C_B) \right\} \quad (9)$$

The three above equations are those integrated by SECAT by a Runge Kutta scheme to calculate the thermal and corrosion performance in the pre-heat region of the boiler. The wall recession rate is calculated by dividing the mass flux of Fe given by Equations (4) and (5) by the Fe density or

$$WR_{mt} = \frac{K\rho_H}{\rho_{Fe}} (C_{mt}^s - C_B) \quad (10)$$

$$WR_{pl} = \frac{K\rho_H}{\rho_{Fe}} (C_{pl}^s - C_B) \quad (11)$$

where

WR = wall recession rate

Fe refers to iron

The correlation used to calculate the heat-transfer coefficients are as follows:

Hg side:

Lubarsky correlation with the provision that Nusselt No. ≥ 7.0

NaK side:

Reference 9 Dwyer's Equation

The mass-transfer coefficient is calculated from the following equation:

$$\frac{KG}{\rho_H} = \frac{.023}{Re^{0.2} Sc^{2/3}} \quad (12)$$

where

G = Hg mass flux

Re = Reynolds number

Sc = Schmidt number

The physical properties are built into SECAT. The diffusion coefficient of Fe in Hg was calculated from the Stokes-Einstein equation. The solubility of Fe in Hg was calculated from the following:

$$C^S(\text{ppm}) = \exp \left[1.217 - \frac{2.113}{T(^{\circ}\text{F})} \right] \quad (13)$$

2. Results

Examination of Equations (10) and (11), the rate of wall recession, reveals that the parameters K, C^S and C_B are controlled by the geometry of the preheat region.

The mass-transfer coefficient (K) is calculated from Equation (12). Substituting for the Reynolds number and collecting terms reveals that K is proportional to $G^{0.8}$. Therefore, it would appear that, if the mass velocity (G) is decreased, a proportionate decrease in the wall recession should result. Let us examine the effect of increasing the thread height by 5%. This 5% increase in thread height increases the flow area by 4.45%. This in turn corresponds to a decrease of 4.26% in G. Finally, this decreased G results in a 3.39% decrease in K. The results in Table 9 show that a 5% increase in thread height yields a 3.2% decrease in maximum tube and plug wall recession rate.

The value of C^S is the saturation concentration of Fe in Hg evaluated at the tube or plug wall temperature. Although the plug wall temperature is at the fluid temperature and therefore defined by the preheat operating conditions, the tube wall temperature is a function of the relation between the individual resistances (tube, NaK and Hg side) and the overall resistance. More specifically, if either the tube or NaK side resistance is increased or the Hg

side resistance is decreased, the C^S and therefore the wall recession rate should decrease. This can be shown by examining the results of Table 9. Increasing NaK jacket diameter decreases the wall recession rate. A larger NaK jacket diameter results in an increase in the NaK side film resistance. The increase in total corrosion is probably due to the longer preheat length required.

If the preheat length is increased by appropriate modification of the geometry, the corrosion will occur over a larger area which should result in

(C_B) a. An increase in the total amount of Fe dissolved in the Hg

b. A decrease in the maximum wall recession rate due to the lowered driving potential for the dissolution of Fe. [The term $C^S - C_B$ of Equations (10) and (11) decreases.]

This supposition is examined by applying it to the results of Table 10, the effect of increasing the number of Hg passages per tube. The model used by SECAT assumes that the heat-transfer area is that portion of the tube in contact with mercury. Since the thread pitch and width are held constant, doubling the number of channels per tube should increase the required preheat length by 24.7%. The results in Table 10 show a length increase of 24.6%. The lesser increase is due to the increased Hg-side heat-transfer coefficient. The combination of increased length and doubling the number of channels results in a net increase in the area exposed to Hg by 34.7%. If the differential equation defining the C_B term is examined (Equation 9), it appears that an approximate solution to it will be of the following form:

$$C_B \approx C^S - [C^S - C_i]e^{-\alpha A}$$

where

C_i = the initial Fe concentration

α = the coefficient involving K and W_H

A = the corrosion area

If the base case results are used, the term αA may be calculated and found to be ~ 0.718 . If it is assumed that the α term is essentially invariant with the number of channels, the value of C_B for an increased (by 34.7%) corrosion area may be calculated. The increase in C_B is found to be 20.4% compared to 19.7% calculated by SECAT. The driving potential and therefore the wall recession rate at the point of maximum wall recession should decrease. Since the driving potential is greater than 1/2 the saturation value, the decrease in maximum wall recession is less than the increase in bulk concentration $\sim 13\%$ decrease compared to a 19.7% increase.

Examination of the results given in Tables 9 and 10 show that the greatest improvement in corrosion performance is obtained by increasing the thread pitch and height. A set of cases was analyzed by SECAT to explore

this supposition. The results shown in Table 11 support this supposition. Further examination of the results revealed that a definite trend was present. This was that improvement in corrosion behavior may be obtained by decreasing the mass velocity of the Hg. This effect is shown in Figure 21 as a plot of the decrease in maximum wall recession rate against the decrease in mass velocity. It should be noted that although the points in Figure 21 are calculated values, they indicate a trend and are not intended to depict a scaleable relationship.

C. MERCURY WETTING TESTS

The program to determine the potential life of 9Cr-1Mo as the SNAP-8 boiler material requires that local wetting as discussed in Section B in the pre-heat and low quality region be achieved to provide the most likely chance that reproducible results will be obtained.

The exact reasons for difficulties in wetting the SNAP-8 boiler can only be conjectural, but it is presumed that relatively small oxide or hydrocarbon layers or absorbed gases are responsible. Wetting is now nearly always achieved by extended contact with flowing Hg at elevated temperatures with the time and temperature apparently a sensitive function of unknown "impurities."

In deriving a wetting procedure, it was recognized that two aspects of the problem exist: (1) cleaning the surface to the point where it can be wet immediately or by a short run in time, and (2) protecting the surface up to the time of Hg injection. Since a principal possibility for a dewetting film occurs from misoperation of the space seal, after the boiler is installed, a procedure in which the surface is cleaned and protected up until first injection is of no value in avoiding in-situ contamination. Therefore, an ideal procedure would be one that could be applied after the boiler is installed and ready to start and could overcome the effects of any preinstallation handling and post startup problems.

Such a procedure was devised, and the principles are as follows:

1. After the boiler is heated and outgassed, the cleaning solution (Hg-based) is forced into the boiler and held for the time and temperature required for wetting
2. The solution is drained or forced by argon pressure back out of the boiler. A film of Hg is left on the wall
3. Hg is injected and presumably wetted operation would be immediately obtained.

Exact procedures must be worked out depending on details of the start-up procedure and requirements to obtain wetting. For example, it may be necessary to rinse the wetted boiler with pure Hg to reduce the active elements in the Hg solution to a low residual level. The great advantage comes if the wetting procedure can be made to fit into the start sequence dictated by present loop and system design and planned tests without major restrictions.

A number of Hg-soluble additives appeared to have the desired activity in removing residual films, including mix-4P3E, and converting them to a harmless Hg-insoluble residue that would precipitate in the Hg vapor portion of the loop. Lithium appeared to be the best choice because it will not alloy with the iron or chromium. Lithium is readily available in pure form, is noncorrosive toward stainless steel up to 1100°F in the times being considered and is easily dissolved into hot Hg. Lithium also has a negligible vapor pressure at SNAP-8 temperatures, so that, if the wetted surface is heated to 1300°F under vacuum in a normal startup, the excess Hg will boil off leaving a very thin layer of Li or Li-Hg compound to protect the surface during this very brief period.

1. Apparatus and Test Procedure

a. Apparatus

The apparatus (Figures 22 and 23) was designed to simulate the SNAP-8 boiler. The 10-in. long capsule, simulating the boiler plug section tubing, was mounted in a tube furnace so that the maximum temperature was located several inches below the top. A thermocouple well, simulating the boiler plug, was inserted from the bottom to about the point of maximum temperature. Mercury was forced in from the bottom to a level (Z-Z in Figure 22) just below the top of the thermocouple well. When the capsule was hot, this arrangement resulted in slow boiling of the Hg and condensing at the cooler top.

The capsule was mounted so that X-rays could be taken to determine the meniscus shape and presence of Hg drops or film on the capsule or thermocouple well surfaces. The rest of the apparatus was arranged to permit evacuating the capsule, introducing argon into the capsule and to regulate the height of Hg in the capsule. The cold trap is used to prevent Hg vapor from entering the vacuum pump. The system can be evacuated to less than 5×10^{-3} torr as measured by a thermocouple gage and could be operated up to 300 psi argon pressure.

A General Electric Model 11HB26 X-ray image amplifier was installed so that the image screen was in the location marked "film" in Figure 22. This assisted in improving the ability to adjust the Hg level and observe the boiling action of the Hg. Permanent record of the meniscus shape was taken with Kodak AA film because resolution with the image amplifier was not good enough to obtain contact angles.

b. Test Procedures

Two series of tests were planned. The first series was to determine the degree of wetting of different combinations of capsules and thermocouple wells with pure Hg. The second series was to be the same as the first except that lithium was to be added to the Hg as a surface cleaning agent. The material combination and corresponding CL-4 test sections are given below:

<u>Capsule</u>	<u>Thermocouple Well</u>	<u>CL-4 Test Section**</u>
9Cr-1Mo	9Cr-1Mo	4A-3, 4A-1, 4B
316 SS	Ta	4A-2
316/clad Cb*	Ta	4A-2

The capsules are reused as required. They are dewetted according to the following procedure: 10% ferrous ammonium sulfate in 10% sulphuric acid, heated to 200°F for 15 minutes, then cleaned by the same method expected to be used for the mass-transfer test sections in the CL-4 tests. The reference surface was then always obtained before each run.

Detailed procedures were finalized and modified as deemed necessary as results were obtained. Modifications to the original apparatus were performed as more details and changes in procedures were required.

2. Results

The runs performed during this reporting period are summarized in Table 12. A brief discussion of the runs is outlined below.

a. Runs 1 through 7

The first 4 runs were made to establish reference conditions with pure Hg and various material combinations, as planned. The objective of the series was to obtain data under reference conditions similar to present SNAP-8 loop conditions. Run 5, with a 9Cr-1Mo capsule, was the first with lithium added. The run was discarded due to a procedural mistake. Run 6 was a repeat after the system was cleaned and refilled. The expected tinning did not occur although some tendency to wet, particularly at lower temperatures at the start, was observed. Wetted contact angles and tinning was not achieved with the 316 SS capsule in Run 7.

b. Runs 8 through 11

The desired overnight exposure at 950°F for 16 hours after vacuum hot out-gassing step (1085°F) was incorporated in Run 8. However, the desired overnight exposure at temperature was not achieved because of a leaky valve. Nevertheless, the thermocouple well was mostly tinned after the run. The surface was not tinned when examined; however, it appears that detinning occurs rapidly after exposure to air. Run 9 was a repeat of Run 6 except that the capsule was vacuum outgassed and held in contact with the Hg-Li solution overnight at 950°F. Complete wetting and tinning was achieved on much of the surface, indicating that the Hg-Li solution can produce the desired result under the right conditions. Run 10 reduced the soak time to 4 hours at 950°F with satisfactory results. Run 11 was a capsule never exposed to mercury before and the same procedure as used in Run 10 and wetting was achieved.

* Cb exposed to the mercury.

** For test section description and other information see Section VI,D.

c. Runs 12 through 14

These runs were made to determine the base line wetting characteristics of 9Cr-1Mo, 316 SS and Cb and Ta when a hot outgassing step is used and no lithium is added to the mercury. Partial wetting was achieved in all runs.

d. Runs 15 through 20

After establishing a basis from the previous experiments, Runs 15 through 20 were made to determine a satisfactory wetting procedure after exposure of the capsule to the Hg-Li mixture and then flushing with two mercury rinses. Satisfactory wetting was achieved from Run 19 and this will be adapted to use on CL-4 and this will be the reference procedure for setting up the apparatus for CL-4.

3. Conclusions

The success of Run 19 indicates that wetting can be attained with two Hg flushes after exposure to the Hg-Li solution with a soaking temperature of 950°F. This will be the reference procedure for setting up the apparatus for CL-4 which is as follows:

- a. Vacuum hot out-gas at 1085°F
- b. Soak with Hg-Li solution at 950°F for four hours
- c. Drain out Hg-Li mixture
- d. Wash with clean mercury-circulate 4 times and drain
- e. Wash with new clean mercury and soak at 950°F for 1 hour
- f. The boiler will then be ready for mercury injection.

The Li concentration of Hg-Li tank and Hg wash will be checked.

It appears that a completely tinned surface can be achieved under suitable conditions to provide for the immediate goals of the Corrosion Loop Program. However, it remains to determine if the solution is effective in cleaning and wetting a surface contaminated by decomposed mix-4P3E.

D. MASS-TRANSFER TESTS - CORROSION LOOP 4

1. Boiler Modification

To run the mass transfer tests on the boiler inlet section to evaluate the proposed corrosion model, a new mercury boiler was designed for CL-4. The new boiler design makes it possible to remove and replace the complete boiler straight inlet section containing the inlet plug without changing the coiled section. A schematic of the 4A boiler is shown in Figure 24.

The 4A boiler was fabricated and installed in CL-4.

2. Removable Test Sections

Four test sections were planned (Table 13) to generate mass transfer data that would be used to evaluate the corrosion model proposed in the parametric analysis (Section B) and the method to reduce the corrosion rate (i.e., reduce the velocity of the liquid in the preheat section). Heat-transfer data from the tests will also be evaluated. The purpose and status of the four test sections are discussed below in the scheduled order of installation in the loop.

a. 4A-2 Test Section

The purpose of this test section was to determine the corrosion pattern in the region where the mercury vapor quality is 5 to 50% to test a postulate made in the corrosion analysis of the boiler. The postulate was that the corrosion after the first generation of vapor was small, since the contact time of the droplets on the wall in the 5 to 50% vapor quality region was short. If this postulate is true, then the main corrosion problem in the SNAP-8 boiler would be confined to the preheat region. Experimental evidence on this point was unclear from previous evaluation of mercury boilers; therefore, a test section was designed that would test the postulate under accelerated conditions.

The 4A-2 test section (see Figure 25) consists of a length of Cb/316 SS bimetallic tubing with the Hg insoluble member (columbium) exposed to the Hg followed by a section of 316 SS between the 5 to 50% mercury vapor quality region. The Hg-insoluble boiler inlet plug is made of Ta. The swirl wire after the inlet plug is also made of Ta.

The purpose of the Hg insoluble inlet plug and inlet tube is to generate two-phase mercury flow in which the liquid contains a very low Fe content. In this way a potential for accelerated mercury corrosion will be provided so that meaningful results will be obtained in a short-term test.

Type 316 SS was selected for the 5 to 50% mercury vapor quality region because its mercury corrosion resistance is lower than 9Cr-1Mo and corrosion effects should be apparent in a short-term test.

The 4A-2 test section was fabricated and installed in the CL-4A boiler. Checkout of the loop was completed and mercury boiling was started on 8 June 1966. The test section exhibited conditioned heat transfer performance immediately upon startup. The section was operated for 140 hours continuously and then was intentionally shut down for replacement of several thermocouples on the test section. The section was restarted after the thermocouple replacement was completed and conditioned performance was again obtained immediately. It is planned to run the 4A-2 section for a total of 300 hours before the section is removed for evaluation.

b. 4A-3 Test Section

The purpose of the 4A-3 section is to check the wetting procedure developed in the mercury wetting tests and to determine the mass-transfer

rate in a boiler inlet plug section fabricated of 9Cr-1Mo. The boiler inlet plug was designed to give a liquid velocity of 4.5 fps in the preheat region of the boiler using a single passage as in the reference SNAP-8 mercury boiler design. A schematic of the section is shown in Figure 26.

The test section was fabricated and will be installed in CL-4 when the testing of the 4A-2 section is completed.

c. 4A-1 Test Section

The purpose of the 4A-1 section is to determine the mass transfer in a 9Cr-1Mo boiler inlet test section when the liquid velocity in the preheat region is 1.1 fps using a multichannel configuration as illustrated in Figure 27.

The 4A-1 section will be fabricated of 9Cr-1Mo steel and the mercury containment tube will be swaged over the boiler inlet plug to reduce the possibility of interpassage flow in the section.

Fabrication tests for machining the multi passage configuration and swaging the tube over the plug were completed. The engineering drawings of the 4A-1 section were completed and the fabrication of the section was started.

d. 4A-4 Test Section

The purpose of the 4A-4 section is to determine the mass transfer in a 9Cr-1Mo boiler inlet test section when the liquid velocity in the preheat region is 0.2 fps and a flashing orifice is used at the end of the preheat region. A schematic of the section is shown in Figure 28.

The engineering drawings of this test section have been completed and the fabrication has started.

3. 4B Boiler

The purpose of this boiler is to determine the potential life of 9Cr-1Mo steel as the mercury containment material in a SNAP-8-type boiler.

The boiler will be fabricated with vacuum-melted modified 9Cr-1Mo steel as the mercury containment material and will be installed in CL-4 after the 4A series of tests is completed. The anticipated operating time of this boiler is 2500 hours. The design of the boiler will be essentially the same as the 4A boiler. The boiler inlet plug design will be based on the results of the 4A test series.

The design of the coiled section of the 4B boiler has been completed and fabrication tests on the coil section have been completed.

E. CORROSION MECHANISM LOOP -1 (CML-1)

A task was initiated during this report period to define the mechanism that controls the corrosion of 9Cr-1Mo steel by liquid mercury in the SNAP-8 boiler under fully wetted conditions. Two mechanisms for 9Cr-1Mo corrosion have been proposed and are as follows:

Mechanism 1 - This mechanism proposes that the corrosion process consists of two phases. In Phase 1 the corrosion is solution rate controlled. In Phase 2 the corrosion is controlled by the diffusion rate of iron through the static Hg contained in channels in the 9Cr-1Mo surface.

Mechanism 2 - This mechanism proposes that the corrosion of 9Cr-1Mo is controlled by the diffusion rate of iron across the liquid laminar boundary layer adjacent to the 9Cr-1Mo surface.

For a given iron concentration gradient, the corrosion rate as defined by mechanism 2 is dependent upon the mercury velocity, while mechanism 1 is not. As compared to 1, mechanism 2 predicts much higher corrosion rates for 9Cr-1Mo in mercury under the flow conditions encountered in the preheat region of the SNAP-8 boiler. In CML-1, three 9Cr-1Mo test samples will be subjected to liquid mercury simulating the flow conditions encountered and bracketing those in the preheat region of the SNAP-8 boiler. Measurement of the depth of corrosion and of the distribution of corrosion from sample to sample will define which of the two proposed mechanisms can be applied to the SNAP-8 boiler design.

The test loop consists of a single liquid-flow system (Figure 29). The mercury is heated to 1100°F and flows through a test section. The 9Cr-1Mo corrosion rate will be measured at the end of the test. The mercury flow-rate is 730 lb/hr to simulate SNAP-8 condition. The mercury is cooled to an acceptable temperature for the pump. Iron buildup is controlled through use of a 2000-lb Hg inventory contained in a tank located at the pump inlet.

The conceptual design of the CML-1 is complete. The following loop components - test section, heater section, and heat-rejection section - have been designed. The mechanical details of the above are being defined. A preliminary plot plan of CML-1 has been made. Available surplus equipment at AGN has been found and designated for use in CML-1. A test cell is being prepared for CML-1. A task to determine the iron concentration in mercury from samples to be collected during the operation of CML-1 has been initiated.

1. Loop

The test loop consists of five major components: mercury pump to flow and pressurize the mercury as required to simulate the SNAP-8 conditions, an electrical heater section to heat the mercury from 500°F to 1100°F, a test section where test specimens of the boiler preheat sections are placed, an air-to-mercury heat exchanger to cool the mercury down to 500°F, and a 2000-lb mercury inventory tank.

Three parameters will be automatically maintained during the tests. These are the mercury temperatures at the test specimens and cooler outlet, and the mercury flow rate through the test specimens.

The mercury temperature in the test specimen will be sensed by a thermocouple and controlled by varying the power input to the electrical heaters. The mercury temperature outlet from the cooler will be sensed by a thermocouple and controlled by varying the air suction pressure to the blower which varies the air flow ratio through the heat exchanger. The mercury flow rate through the test specimen will be sensed by a venturi meter and controlled by a flow control valve.

2. Components

a. Test Section

Several test section configurations were considered for use in the CML-1 involving series and parallel samples and single- and multi-test concepts. The one finally selected was three 9Cr-1Mo tubes in a series of different flow channel diameters to be run in a single test. These samples are short (1/2 in. long) pieces of 9Cr-1Mo tubing in a bimetallic stainless steel and columbium carrier. Columbium is used to preclude significant iron pickup from regions other than the 9Cr-1Mo sample. The largest-diameter tube is placed at the inlet and the smallest at the outlet. The Cb carrier is sized to provide an inlet section of approximately 20 diameters to establish the flow pattern in the mercury before it reaches the 9Cr-1Mo samples. Figure 30 and Table 14 are sketches and specifications for the test sections. Figure 31 depicts the predicted corrosion behavior of the test sections under presumed applicability of mechanism 2 type corrosion. The analytical treatment appears in Reference 11.

The channel configuration selected for the test sample is a round tube. The significant flow conditions in the preheat region of the SNAP-8 boiler are as follows:

Reynolds number = 57200

Hg velocity = 4.5 fps

For a round tube, given the above parameters, it follows that the flow rate and flow channel diameter are uniquely defined. The flow rate is 729.8 lb/hr and the diameter 0.104 in.

From the requirement that the other two 9Cr-1Mo samples bracket the flow condition given above, it is apparent that if the test samples are arrayed in a parallel manner, the total loop mercury flow rate will be approximately 2000 lb/hr. However, if the samples are tested in three different runs or arrayed in series for a single run, the total flow will be 730 lb/hr. If the mercury test temperature is 1100°F, the corresponding power requirements for the parallel and series-sequential arrangements are 15 and 5 kw, respectively. In addition, parallel configuration dictates the need to either calculate the mercury flow rate in each sample or provide three separate flowmeters. The only advantage with a parallel arrangement is that all test samples are exposed to the identical mercury

chemistry at the inlet. However, for a sample of reasonable length (approximately 1.0 in.), the iron pickup across the test sample for the expected corrosion rate (approximately 5 mils/100 hr as predicted under mechanism 2) is less than or equal to 0.01 ppm. Therefore, for the above reasons the parallel concept was eliminated from further consideration.

If the sequential test concept is employed, there exists a potential danger that identical conditions cannot be maintained for the three test runs. This would make evaluation and comparison of the test data difficult, if not impossible. Therefore, the series sample single-test concept was selected for CML-1 test section.

The middle (base) test sample has been defined previously. The bracketing samples are arbitrarily selected to operate at 2x and 1/2x the velocity of the base sample. Since a series concept has been selected, the mercury mass flow rate is identical in all the test samples. The sample flow diameters are, therefore, set and are equal 0.147, 0.104, and 0.074 in. There remains to be defined the sample length and the mercury inventory.

A parametric study was performed to determine the depth of wall recession as a function of sample length and mercury inventory. Mercury inventory is involved in the study since this is the manner in which iron buildup in the mercury is controlled. The results were used to determine the specifications for the test section given in Table 14. The design point was selected as the one giving measurable wall recession in all three samples requiring a reasonable mercury inventory and having a reasonable sample length.

The pressure drop across the test section was calculated using the Blasius correlation for the friction factor and a standard reference text for the evaluation of expansion and contraction losses.

b. Heater Design

The heater operating conditions are tabulated below:

Mercury flow rate	730 lb/hr
Heater inlet temperature	500°F
Heater outlet temperature	1100°F
Heat load (net to fluid)	4.2 kw

A Cb-lined tubing of 0.51 in. OD will be used in all high-temperature sections of the loop; this would include the heater section. Electrical clam-shell heaters will be used for heating the mercury. The HEVI-DUTY electric heating units are rated to 2200°F; for long heater element life, its temperature will be limited to 1800°F for this design.

A simple radiation heat-transfer analysis indicated that more than 10 ft of heater length would be required to heat the mercury in the tube to the desired temperature. It is highly desirable to decrease its length so that

a single straight length of the Cb-lined tubing can be used. This tube cannot be bent in a sharp radius. The test cell size is 8 ft long, 6 ft wide and 10 ft high.

Since the controlling heat-transfer mode is by radiation, the only way to shorten the heater length is to package the required heat-transfer area in a shorter length. This can be done by slipping a metal sleeve around the mercury tubing to increase its surface area in contact with the heater per unit length. A strong, double pipe with a nominal diameter of $3/4$ in. (1.05 in. OD and 0.434 in. ID) can be used for the sleeve by reaming out the ID to 0.51 to fit over the Cb-lined tubing. The presence of the sleeve has created two additional heat flow resistances - the sleeve itself and the gap between the sleeve and tubing.

By limiting the heater length to 6 ft, it can be placed horizontally on the 8-ft-long wall with sufficient room for the large radius bend of the Cb-lined tubing to a vertical test section and for insulation at the ends of the heater. The results of the heater thermal analysis (Reference 11), along with the assumptions made, follows.

The Hg-side heat-transfer coefficient was computed as 1800 Btu/hr-ft²-°F for an average film drop of 12°F. Since the electrical heater power will be constant over its length, the heat flux per unit length will be assumed constant. The temperature drop across the tubing was neglected since it was computed as less than 10°F.

Assuming that the sleeve can be placed on the tubing with a radial clearance of 0.002 in., this value was used to compute the temperature drop across the gas gap as 92°F. It is desirable to minimize this gap as much as possible. The sleeve can be cut in short axial lengths to minimize the required clearance for assembly.

The temperature drop across the metal sleeve was computed as 29°F. The thermal conductivity was taken as 10 Btu/hr-ft-°F which is typical for stainless steel.

The radiation heat transfer from the clam shell heaters to the sleeve was computed assuming an emissivity of 0.5 for both the heater and sleeve surfaces. The maximum computed temperature was 1690°F.

The predicted temperatures of the heater, sleeve and tubing at the heat inlet and exit are shown on the following page.

1510°F	Clam Shell heaters	1690°F
--------	--------------------	--------

664	SS Metal Sleeve	1241
635		1202

510	Cb-Lined Tubing	1110
-----	-----------------	------

500 Mercury Flow —————> 1100

TUBE ϕ —————

The clam shell heaters will be insulated by one inch of Fiberfrax and 3 inches of Johns-Manville Superex pipe insulation on the outside. The latter is rigid and can support the weight of the heater sleeve, tubing and Hg. The insulated heater assembly will be supported at the bottom over its entire length in a 10 inch channel-iron beam.

The heat loss from the heater assembly through the insulation to ambient was computed as approximately 1 kw (Reference 11). Thus the heat loss to ambient is approximately 20% of the heater power. The corresponding heater power during operation would be approximately 5.6 kw. The calculated outside surface temperature of the heater insulation is 164°F.

c. Cooler Design

The cooler operating conditions are tabulated below:

Mercury flow rate	730 lb/hr
Mercury inlet temperature	1100°F
Mercury outlet temperature	500°F
Heat Load	4.2 kw

It is proposed that an existing Clarage blower be used for the cooler. This blower has a pressure rise of 8 in. of water at 1883 rpm. Flow data down to 1600 cfm are available. No surge was detected down to this flow rate. For an air bulk rise of 50°F, the air flow rate must be approximately 310 cfm. Therefore, approximately 80% of the blower output must be bypassed around the blower.

A counter-flow air-mercury heat exchanger was designed. The mercury flows in a 0.51 OD Cb-lined tubing. When the heat exchanger was sized with an 0.51 ID air flow annulus, the length was approximately 9 ft long and has a pressure drop of approximately 6 in. of water. This heat exchanger is too long to be placed in the test cell. Thus, a metal sleeve was placed around the Cb-lined tubing as was done in the heater design.

The heat exchanger design must satisfy both the heat-transfer and pressure-drop requirements. Assuming that the heat exchanger air inlet temperature is 100°F, the effectiveness is 0.60. For a counter-flow heat exchanger with the fluid bulk temperature change of 50°F and 600°F, respectively, for the air and mercury, the effectiveness was computed as 0.62 at an NTU* of one (Reference 11). Thus, using NTU of 1.0 in the design would be slightly conservative. The required overall conductance was computed as 24 Btu/hr-°F. The following equation satisfies the heat-transfer requirements (see Reference 11 for derivation).

$$\frac{1}{24} = \frac{(D_o^2 - D_1^2)}{73.8(D_o + D_1) \cdot 2 D_1 L W^{.8}} = \frac{0.0670}{L}$$

where

D_o = air flow annulus OD, in.

D_1 = air flow annulus ID, in.

L = heat exchanger length, ft

W = air flow rate, lb/sec

The above equation was based on the following assumptions:

- (1) The heat flow resistance through the Cb-lined tubing was based on the thermal conductivity of stainless steel which is smaller than for Cb.
- (2) A 0.002 in. radial air gap assumed between the tubing and the sleeve ID.

The air pressure drop through the heat exchanger must be limited to approximately 5.5 in. of water. A total of 8 in. of water pressure head is available but some losses will be taken through the ducting from the blower to the heat exchanger and from the heat exchanger to exit vent. The following equation (Reference 11 includes the derivation) expresses the total pressure drop through the heat exchanger.

$$\Delta P_\gamma = \left[1.7 + .138 \frac{(D_o + D_1) \cdot 2 L}{(D_o - D_1) W^{.2}} \right] 1510 \frac{W^2}{(D_o^2 - D_1^2)^2}$$

where ΔP_γ = total heat exchanger pressure drop, in. of water.

*UA/WC_P.

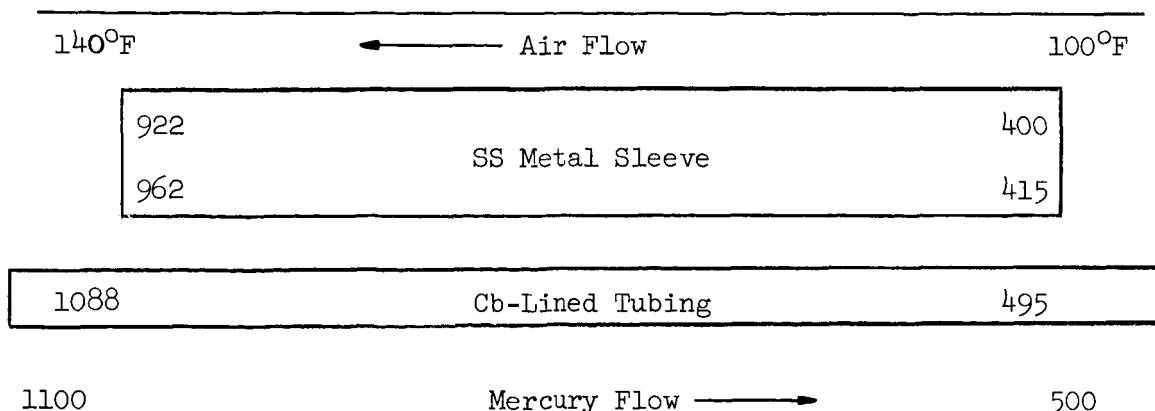
The preceding equation includes the entrance and exit loss using loss coefficients of 0.5 and 1.0, respectively. The acceleration pressure drop was based on a 50°F air bulk temperature rise and a pressure drop of 5.5 in. of water. The Fanning friction factor of $0.0461R_e^{-.2}$ was used in the analysis.

The air flow rate does not have to be the one which corresponds to the 50°F bulk rise assumed in the analysis thus far. Both the NTU and acceleration pressure drop terms are insensitive to variation in flow rate a factor of 2. The first equation was solved for the flow rate, w , and substituted into the second to give the following:

$$\Delta P = \left[1.7 + 0.404 \frac{D_1^{3/4} L [0.0417L - 0.0656]^{1/4}}{(D_o - D_1)^{1.25}} \right] \times 0.0323 \frac{(D_o - D_1)^{1/2}}{D_1^{2.5} [0.0417L - 0.0670]^{2.5}}$$

The inside diameter of the flow annulus, D_1 , is fixed at 1.05 in. The annulus OD must be the ID of a standard pipe. For a 3-in. nominal schedule 5 pipe (3.334 in. ID) and a heat exchanger length of 6.5 ft, the total pressure drop was computed as 5.4 inches of water. The flow rate was computed as 0.394 lb/sec from the second equation. The air bulk rise was computed as 40°F which is close enough to the 50°F assumed that the NTU of one used in the analysis is still conservative.

The air duct pressure drop was computed assuming that the total duct length is 50 ft and has a total of 8-90° turns. The duct diameter of 6 in. was selected. The duct pressure drop was computed as 0.32 in. of water. Thus, the total blower head required is 6.5 in. of water. The predicted temperatures of the Cb-lined tubing and sleeve at the entrance and exit are shown below.



TUBE ϕ —————

d. In-House Equipment

The mercury Chempump that will be used for this loop was used in CTL-2 in the same test cell location. It is equipped with a bypass flow control complete with valves and pressure gages. The pump and associated plumbing will be used as is. This pump is rated at 0.5 gpm flow rate at a pressure head of 88 ft.

Two Clarage blowers are available and are located on top of the test cells. One of these blowers will be used in the air-mercury cooler. Test data from this blower is available at a speed of 1883 rpm and flow rate of 1800 cfm to 400 cfm; the blower pressure head at the exit was measured as 8 in. of water to 3.5 in. at the lowest and highest flow rate, respectively.

A Fox venturi is available to sense the mercury flow rate through the test section. The valve positioner and D/P cell used in the previous loop will be used for this loop.

3. Mercury Analysis

The corrosion behavior of the test samples was calculated assuming that the iron dissolved from the 9Cr-1Mo specimens remained in the mercury stream. However, if a portion of the iron in the mercury is trapped somewhere in the loop the total wall recession shown in Figure 31 would be too low. This is due to a higher-than-calculated concentration gradient in the test section region which in turn increases the 9Cr-1Mo corrosion rate. Therefore, the evaluation of the post-test data will require a knowledge of the iron concentration in the mercury at the inlet to the test section. A procedure is being set up whereby an iron concentration in a mercury sample may be determined. The procedure consists of a vacuum distillation of the mercury sample followed by a spectro-photometric analysis of the residue. Mercury samples at the inlet to the heaters will be collected periodically during the test run.

REFERENCES

1. H. Derow and B. E. Farwell, SNAP-8 Materials Report for July-December 1965, Aerojet-General Report No. 3134, February 1966.
2. H. E. Bleil, Evaluation of Indium-Containing 316 Stainless Steel Thermowells from PCS-1 Phase IV, Aerojet-General Technical Memorandum TM 4923:66-425, 13 July 1966.
3. Cleaning Mercury-Contaminated Components, Procedure for, Aerojet-General Specification AGC-10319/10A, 19 May 1966.
4. SNAP-8 Electrical Generating System Development Program, Monthly Progress Report No. L0740-05-17, Aerojet-General Corporation, May 1966.
5. H. E. Bleil, Evaluation of PH 15-7 Mo for TAA Second Stage Shroud, Aerojet-General Technical Memorandum TM 4921:66-419, 29 June 1966.
6. Brazing Technical Bulletin No. T-3, Handy and Harmon, New York, New York.
7. J. N. Hodgson, L. B. Kelly, and A. H. Kreeger, Performance Analysis on the -1 Boiler Conditioning (RPL-2), Aerojet-General Technical Memorandum TM 4833:64-8-259, 18 December 1964.
8. Design Report on Dynamic Corrosion Loops for the SNAP-8 System, Aerojet-General Report No. 2596, June 1963.
9. H. Derow and B. E. Farwell, SNAP-8 Materials Report for January-June 1965, Aerojet-General Report No. 3038, July 1965.
10. A. J. Sellers, SNAP-8 Tube-In-Tube Boiler Design Analysis, Aerojet-General Technical Memorandum TM 4803:65-2-223, February 1965.
11. D. Yee and L. A. Kimura, CML-1 Design Report, Aerojet-General Technical Memorandum TM 4923:66-439.

TABLE 1

NaK GAS-FIRED HEATER WALL THICKNESS ANALYSIS SUMMARY

Item	No. 1 Heater	No. 2 Heater
1. Average measured wall thickness (mils) 6/29/65 5/13/66	206.5 198.9	208.6 195.7
2. Average wall thinning (mils)	7.6	12.9
3. Operating time between 6/29/65 & 5/13/66 (hr)	970	970
4. Average wall thinning rate (mils/1000 hr) - Item 2 (1000) Item 3	7.8	13.3
5. Statistically determined minimum wall thick- ness (mils)* 6/29/65 5/13/66	178 167	177 164
6. Min. wall thickness actually measured (mils) 6/29/65 5/13/66	190 175	190 175
7. Wall thinning rate based on Item 6 (mils/1000 hr)	15.4	15.4
8. Remaining safe operating life** (hr)		
a. Using wall thinning rate (Item 4) deter- mined from average of measurements	12,600	7,210
b. Using wall thinning rate (Item 7) deter- mined from min. of actual measurements	6,420	6,230

* A statistical analysis of actual measurements indicates that 99.9% of the wall is equal to, or greater than, the presented value; and there is an associated 99% confidence level.

** The time required for the NaK containment tube wall thickness to be reduced to the boiler code minimum allowable wall of 0.068 in. at a pressure of 75 psi and a temperature of 1500°F. The present wall thickness is presumed to be the statistically determined minimum (Item 5) on 5/13/66.

TABLE 2

ORGANIC ANALYSIS OF Hg DUMP-TANK SAMPLES FROM PCS-1 PHASE IV STEP 2

Date Sample Taken	Sampling Location	Total Sample Weight (grams)	Weight of Organic (grams x 10 ⁻³)		Weight of Organic to Sample Weight (ppm)	
			Mix-4P3E	CH	Mix-4P3E	CH
1-25	Subsurface Main Tank Prior to Step 2	-	ND	ND	ND	ND
1-31	Subsurface Emergency Tank	21,077	0.45	0.07	0.02	0.003
2-1	Surface Emergency Tank	389	0.24	0.7	0.62	1.8
2-1	Surface Main Tank	1,434	0.06	0.08	0.04	0.06
2-4	Surface Main Tank	1,017	0.50	0.92	0.49	0.9
2-4	Surface Emergency Tank	654	ND	1.14	<0.01	1.7
2-14	Surface Main Tank	527	0.04	1.04	0.08	1.97
2-14	Surface Emergency Tank	529	0.3	0.82	0.56	1.6
3-16	Surface Main Tank	881	Trace	1.06*	<0.01	1.20*
3-16	Surface Emergency Tank	322	Trace	0.6*	<0.01	1.86*
4-1	Surface Main Tank	1,178	0.14	0.24	0.12	0.20
4-1	Surface Emergency Tank	663	ND	0.17	ND	0.26
4-13	Surface Main Tank	1,223	ND	0.14	<0.01	0.11
4-13	Surface Emergency Tank	551	3.5	2.84	6.4	5.1

Hg from main tank after it was drained:

Subsurface	641	ND	<0.03	ND	<0.05
Surface	34,050	13.5	4.9	0.4	0.14

Trace = Detected but less than 0.001 ppm.

ND = Not detected at a detection limit of 0.005 ppm.

* This was an ester or carbonyl which masked out the CH (Aliphatic hydrocarbon) if there was any present.

TABLE 3

FLUIDS ANALYSIS FOR Na, K, AND Hg

<u>Sample Material and Location</u>	<u>Date Samples Taken</u>	<u>Sample Wt (grams)</u>	<u>Na Wt (mg)</u>	<u>K Wt (mg)</u>	<u>Na+K Sample Wt (ppm)</u>	<u>K/Na Ratio</u>
Hg from emergency dump tank	4/13/66	551	0.08	0.26	.61	77/23
Hg from top of main dump tank	4/18/66	1426	0.010	0.037	.033	79/21
Hg from emergency dump tank	2/1/66	1522	0.025	0.037	.041	60/40
Hg - Reprocessed	--	1415	0.012	0.00	.008	00/100
Mix-4P3E from L/C dump tank	4/18/66	84.7	0.050	0.00	.59	00/100
			<u>Hg Wt (g)</u>	<u>Hg/NaK (ppm)</u>		
NaK from HRL	4/18/66	4.8	3	0.6		
NaK from HRL	4/19/66	5.1	5	1.0		
NaK from PNL	4/18/66	7.4	6	0.8		
NaK as received from vendor	--	12.0	17.5	1.5		

TABLE 4

MIX-4P3E SAMPLE ANALYSIS - PCS-1 PHASE IV

Source of Sample	Infrared Spectro- photometric Analysis	Viscosity, Centipoises		Specific Gravity	Refractive Index	Phenole (wt%)	Isomer Distribution** (wt%)			
		100°F	210°F				mm+ 4P3E	mp- 4P3E	mo+ oo- 4P3E	pp- 4P3E
TAA space-seal trap	Typical mix-4P3E spectrogram	70.3	6.26	1.1763	1.6203	0.046	54.0	38.0	4.3	3.7
Alternator cavity drain trap	Typical mix-4P3E spectrogram with evidence of vacuum pump oil*	66.2	6.60	1.1749	1.6201	0.046	58.7	33.1	4.9	3.3
Dump tank	Typical mix-4P3E spectrogram	66.9	6.29	1.1754	1.6200	0.042	56.5	34.4	5.4	3.7
L/C vacuum-pump trap	Typical mix-4P3E spectrogram with considerable amount* of vacuum pump oil	--	--	--	1.4790	--	--	--	--	--
Control (P.O. 106587 manufactured 12/11/64)	Typical mix-4P3E spectrogram	66.0	5.95	1.1740	1.6199	0.071	52.5	39.1	5.3	3.1

* Roughing-pump-type oil equivalent to "Duoseal" oil.

** Isomer definitions:

op-4P3E: ortho-para phenoxyphenyl ether
 mm-4P3E: meta-meta phenoxyphenyl ether
 mp-4P3E: meta-para phenoxyphenyl ether
 mo-4P3E: meta-ortho phenoxyphenyl ether
 oo-4P3E: ortho-ortho phenoxyphenyl ether
 pp-4P3E: para-para phenoxyphenyl ether

TABLE 5
CHEMICAL ANALYSIS OF FILINGS FROM SNAP-8 ALTERNATOR WELD AREAS
AFTER 140 HOURS OF OPERATION IN PCS-1 PHASE IV STEP 2 TESTS

Element	HY80	MIL-S-16216 HY80	Mild Steel Case	Original Weld Remote from Trunnion		Original Weld at Uncracked Trunnion	Mild Steel Weld Repair	Inconel A Specifica- tion
				Sample 1	Sample 2			
Fe	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	6/12.0
Ni	2.80	2.0/3.25	0.25	1.61	1.75	1.67	0.64	Bal.
Cr	2.55	1.0/1.80	0.31	1.05	0.83	0.91	0.58	13.0/17.0
Mn	0.46	0.10/0.40	0.77	0.48	0.58	0.57	0.95	1.0/3.5
Mo	0.50	0.20/0.60	0.09	0.30	0.21	0.22	0.15	0.50/2.0
O	ND	0.18 max.	ND	0.26	ND	ND	ND	0.5 max.

ND = Not Determined.

TABLE 6

ALTERNATOR SEAL WELD CONFIGURATIONS AND RESULTS OF CHEMICAL SPOT TESTS OF WELDS

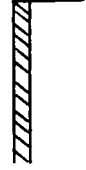
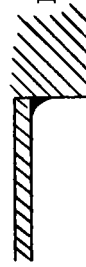



Alternator	Seal Weld Configuration		Chemical Spot Test Analysis of Seal Welds	
	At Trunnion Boss	Away from Trun. Boss	At Trunnion Boss	Remote From Trunnion Boss
S/N 481489 (Seal weld cracked in PCS-1 operation.)	mild steel 	Not sucked back. Looks as if filler was added.	Configuration prevented testing. (Fe base by emission)	Iron base alloy
S/N 481491 (Installed in PCS-1 Phase IV for continued Step 2 tests)	mild steel 	Some suck-back. Looks as if very little, if any, filler was added.	Nickel base alloy	
S/N 481510 (Being built up in Clean Room)	mild steel 	Sucked back as if no filler was added.	Nickel base alloy	
S/N 481490	mild steel 	Sucked back as if very little or no filler was added.	Iron base alloy	
S/N 481492	mild steel 	Sucked back as if very little or no filler was added.	Configuration prevented testing.	Iron base alloy

Table 6

TABLE 7SNAP-8 -1 BOILER⁽¹⁾ PERFORMANCE HISTORY IN RPL-2

Operating Period	July 1964 to June 1965
NaK Side	
Total operation	2350 hr ⁽²⁾
Nominal boiler design conditions	
Flow (lb/hr)	3200
Temperature (°F)	
Inlet	1300
Outlet	1100
Hg Side	
Total operation	1415 hr
With rubidium	320 hr (Nov. 1964 to Mar. 1965)
Nominal boiler design conditions	
Flow (lb/hr)	11,400
Temperature (°F)	
Inlet	513
Outlet	1265
Pressure (psia)	
Inlet	340
Outlet	270
Vapor Outlet conditions (typical of various test periods)	
90% quality	470 hr ⁽³⁾
Saturated vapor	240 hr
9 ft superheat length	125 hr
27 ft superheat length	460 hr
42 ft superheat length	120 hr

⁽¹⁾P/N 092020-1F S/N A-1.⁽²⁾Approximately the last 300 hr included a NaK purification system.⁽³⁾Flow was 50% of nominal to enhance conditioning.

TABLE 8

DEPTH OF SURFACE DEFECTS ON BAR AND WIRE OF
Hg INLET PLUG FROM -1 BOILER

Distance from Hg Inlet End (ft - in.)	Wire to Wire Pitch (in.)	Defect Depth (mils)	
		Bar	Wire
0-5	1-1/2	0	1.7 (Pit)
1-4	1-1/2	0.8 (Pit)	4.2 (Pit)
2-7	1-1/2	0.8 (Pit)	ND
5-9	2-1/2	0	0
9-1	2-1/2	0.4 (Crevice)	2.5 (Pit)

ND - Not determined

TABLE 9

EFFECT OF GEOMETRY ON THE PERFORMANCE CHARACTERISTICS IN
THE PREHEAT REGION OF THE SNAP-8 BOILER

Variable	Percent Change ⁽¹⁾ in Performance Characteristics for						
	5% Increase of Variable			5% Decrease of Variable			
	Maximum Penetration	Total Corrosion	Preheat Length	Hg Side ΔP	Maximum Penetration	Total Corrosion	Preheat Length
NaK Jacket ID	-1.4	+1.6	+2.8	+2.8	+1.6	-1.7	-3.0
Hg Tube ID ⁽³⁾	-0.11	+0.04	-5.0	~0	+0.27	-0.16	+5.2
Thread Height	-2.9	-2.0	+0.69	-12	+3.1	+2.1	-0.71
Thread Pitch	-2.2	-3.0	-0.49	-15	+2.3	+3.3	+0.58
Thread Width	+0.40	+0.54	+0.92	+2.9	-0.36	-0.54	-0.91
							-3.3

NOTES:

- (1) The change in the performance characteristics is that compared to the values calculated for the base geometry. The base geometry is that described in Reference 10.
- (2) Total corrosion is the amount of wall material dissolved in the mercury.
- (3) The corresponding OD is changed to maintain a tube wall thickness of 0.09 inches.

TABLE 10

EFFECT OF THE NUMBER OF CHANNELS PER TUBE ON THE
PERFORMANCE CHARACTERISTICS IN THE PREHEAT REGION OF
THE SNAP-8 BOILER AT A CONSTANT MASS VELOCITY (G)

No. of Channels per Tube	Percent Change in Performance ⁽¹⁾ Characteristics			
	Maximum Penetration	Total Corrosion ⁽²⁾	Preheat Length	Hg Side ΔP
2	-12	+18	+24	+34
3	-31	+45	+61	+116
4	-58	+72	+133	+370
5	-84	+82	+338	~15X
} (3)				
2	-5.4	+10	+15	+19
3	-10	+19	+29	+40
4	-15	+27	+43	+61
5	-19	+33	+57	+83
} (4)				

(1) The change in the performance characteristics is that compared to the values calculated for the base geometry. The base geometry is that described in Reference 10.

(2) Total corrosion is the amount of wall material dissolved in the mercury.

(3) Constant mass velocity (G) maintained by increasing thread height.

(4) Constant mass velocity (G) maintained by increasing thread pitch.

TABLE 11EFFECT OF THREAD HEIGHT AND PITCH ON THE PERFORMANCE
CHARACTERISTICS IN THE PREHEAT REGION OF THE SNAP-8 BOILER

Thread Height (in.)	Performance Characteristic	Percent Change ⁽¹⁾ in Performance Characteristics Thread Pitch (in.)					
		.375	.75	1.125	1.5	1.875	2.25
.062	Maximum Corrosion Rate	(3)	-29	-42	-49	-54	-57
.082		-16	-42	-53	-60	-63	-66
.102		-27	-51	-61	-66	-69	-71
.122		-36	-57	-66	-71	-74	-76
.062	Total ⁽²⁾ Corrosion	(3)	-34	-47	-53	-57	-59
.082		-11	-44	-55	-60	-63	-65
.102		-18	-49	-60	-64	-67	-68
.122		-24	-54	-63	-67	-70	-71
.062	Preheat Length	(3)	-2.9	-2.1	-1.0	-1.0	+.60
.082		+4.4	+3.3	+5.3	+7.2	+8.7	+9.9
.102		+8.6	+9.3	+13	+16	+18	+19
.122		+12	+15	+18	+24	+27	+29
.062	Hg Side ΔP	(3)	-88	-96	-98	-98	-99
.082		-54	-94	-98	-99	-99	-99
.102		-74	-99	-99	(4)	(4)	(4)
.122		-84	-98	-99	(4)	(4)	(4)

NOTE:

- (1) The change in the performance characteristics is that compared to the values calculated for the base geometry. The base geometry is that described in Reference 10.
- (2) Total corrosion is the amount of wall material dissolved in the mercury.
- (3) Base case.
- (4) Greater than 99.5% decrease.

TABLE 12
SUMMARY OF CAPSULE WETTING TESTS

Run No.	Material		Fluid	Max. Temperature of Exposure, °F	Remarks
	Capsule	Thermocouple Well			
1	9Cr-1Mo	9Cr-1Mo	Hg	1085*	No wetting observed from X-ray and visual observation after test
2	316 SS	316 SS	Hg	1085	Same as above
3	9Cr-1Mo	Ta	Mercury	1085	Same as above
4	316/Cb Tubing; Cb exposed to the mercury	Ta	Mercury	1085	Same as above
5	9Cr-1Mo	9Cr-1Mo	Hg with approx. 550 ppm lithium	--	Incomplete test; redone in Run 6
6	9Cr-1Mo	9Cr-1Mo	Same as above	1085	Spotty wetting on thermocouple well. X-ray and visual examination made after test.
7	316 SS	316 SS	Same as above	1085	No wetting observed from X-rays or visual examination.
8	9Cr-1Mo	9Cr-1Mo	Hg-550 ppm of Li	--	Wetting of tubing and thermocouple well was achieved after the capsule was outgassed at 1085°F and the Hg-Li solution was held in the capsule for 16 hr at 950°F.

After Run 8, the capsule wetting apparatus was cleaned and a new Hg-Li solution was added because of an accident during operation of the apparatus.

(cont.)

*Time of exposure for Runs 1 through 7 was 1 hour.

TABLE 12 (cont.)

Run No.	Material		Fluid	Remarks
	Capsule	Thermocouple Well		
9	9Cr-1Mo	9Cr-1Mo	Hg-454 ppm of Li	Capsule and thermocouple well from Run 8 were dewetted and cleaned prior to the test. Wetting was achieved using the same procedure that was used in Run 8.
10	9Cr-1Mo	9Cr-1Mo	Hg-454 ppm of Li	Capsule and thermocouple well from Run 9 were dewetted and cleaned prior to the test. Wetting was achieved after the capsule was outgassed at 1085°F and the Hg-Li solution was held in the capsule for 4 hr at 950°F.
11	9Cr-1Mo	9Cr-1Mo	Hg-454 ppm of Li	A new 9Cr-1Mo capsule and thermocouple well were installed for the test. Wetting was achieved using the same procedure that was used for Run 10.
12	9Cr-1Mo	9Cr-1Mo	Hg	This test was run to determine the base line wetting characteristics of 9Cr-1Mo when a hot outgassing step is used and no Li is added to the mercury. Partial wetting of the thermocouple well and capsule was observed.
13	316 SS	316 SS	Hg	This test was run to determine the base line wetting characteristics of 316 SS when a hot outgassing step is used and no Li is added to the mercury. Partial wetting was achieved.
14	316 SS/Cb	TA	Hg	This test was run to determine the base line wetting characteristics of Cb and Ta when a hot outgassing step is used and no Li is added to the mercury. Partial wetting of the Ta and Cb was observed.
15	9Cr-1Mo	9Cr-1Mo	Hg-527 ppm Li	Capsule and thermocouple well were dewetted and cleaned prior to the test. The capsule was hot outgassed, exposed to the Hg-Li solution for 1 hr at 1085°F and 4 hr at 950°F. The Hg-Li mixture was drained from the capsule and replenished with new mercury. The capsule was then heated to 950°F for 1 hr. The capsule and thermocouple well appeared wetted.

(cont.)

TABLE 12 (cont.)

Run No.	Material		Fluid	Remarks
	Capsule	Thermocouple Well		
16	9Cr-1Mo	9Cr-1Mo	Hg-637 ppm Li	Capsule and thermocouple well were dewetted and cleaned prior to the test. This run was made to test a proposed wetting procedure for CL-4. The capsule was hot outgassed and exposed to the Hg-Li solution for 4 hr at 750°F. The Hg-Li mixture was drained out and replenished with new mercury. The capsule and thermocouple well were wetted.
17	9Cr-1Mo	9Cr-1Mo	Hg-503 ppm Li	Capsule and thermocouple well were dewetted and cleaned prior to the test. This run was made to attempt to duplicate the success of Run 16 except that two Hg flushes were made after exposure to the Hg-Li solution. The capsule was hot outgassed and exposed to the Hg-Li solution for 4 hr at 750°F. The Hg-Li mixture was drained out and replenished with new mercury, circulated and drained. New mercury was then added and the capsule was then heated to 750°F for 1 hr. Spotty wetting on the capsule and thermocouple well was achieved. Not satisfactory for wetting purpose.*
18	9Cr-1Mo	9Cr-1Mo	Hg-473 ppm Li	Capsule and thermocouple well were dewetted and cleaned prior to the test. This run duplicated Run 17 in procedure to ensure the results obtained and check out and have a base line for the modifications. Spotty wetting on the thermocouple well and capsule was achieved. Not satisfactory for wetting purpose.

(cont.)

* After Run 17 minor modifications were performed.

TABLE 12 (cont.)

Run No.	Material		Fluid	Remarks
	Capsule	Thermocouple Well		
19	9Cr-1Mo	9Cr-1Mo	Hg-304 ppm Li	Capsule and thermocouple well were dewetted and cleaned prior to the test. This run was made to test a proposed wetting procedure for CL-4. The capsule was hot outgassed and exposed to the Hg-Li solution for 4 hr at 950°F. The Hg-Li mixture was drained out and replenished with new mercury, circulated and drained. New mercury was then added and the capsule was then heated to 950°F for 1 hr. The capsule and thermocouple well were wetted.
20	9Cr-1Mo	9Cr-1Mo	Hg-302 ppm Li	Capsule and thermocouple well were dewetted and cleaned prior to the test. This run duplicated Run 19 except the temperature was 850°F instead of 950°F. This was for both the Hg-Li solution and the new mercury flush. Spotty wetting on the thermocouple well and capsule was achieved. Not satisfactory for wetting purpose.

TABLE 13
SUMMARY OF REMOVABLE STRAIGHT SECTIONS - 4A BOILER

<u>Designation</u>	<u>Design Characteristics of Boiler Inlet Plug</u>	<u>Material of Construction</u>
4A-2	Single passage 4.5 fps liquid velocity in the preheat region.	Cb-lined mercury containment tubing in the preheat region. Ta boiler inlet plug and turbulator wire. 316 SS-Hg containment tubing in the 5 to 50% vapor quality region.
4A-3	Single passage 4.5 fps liquid velocity in the preheat region. (Reference SNAP-8 boiler design.)	All mercury wetted surfaces 9Cr-1Mo steel.
4A-1	Multipassage - 1.1 fps liquid velocity in the preheat region.	All mercury wetted surfaces 9Cr-1Mo steel.
4A-4	Single passage - 0.2 fps liquid velocity in the preheat region. Flashing orifice at the end of the preheat region.	Orifice-tungsten. End of plug after the orifice-Stellite 6B. All other parts wetted by the Hg-9Cr-1Mo steel.

TABLE 14

GEOMETRIC AND PERFORMANCE PARAMETERS FOR THE CML-1 TEST SECTION

Geometric

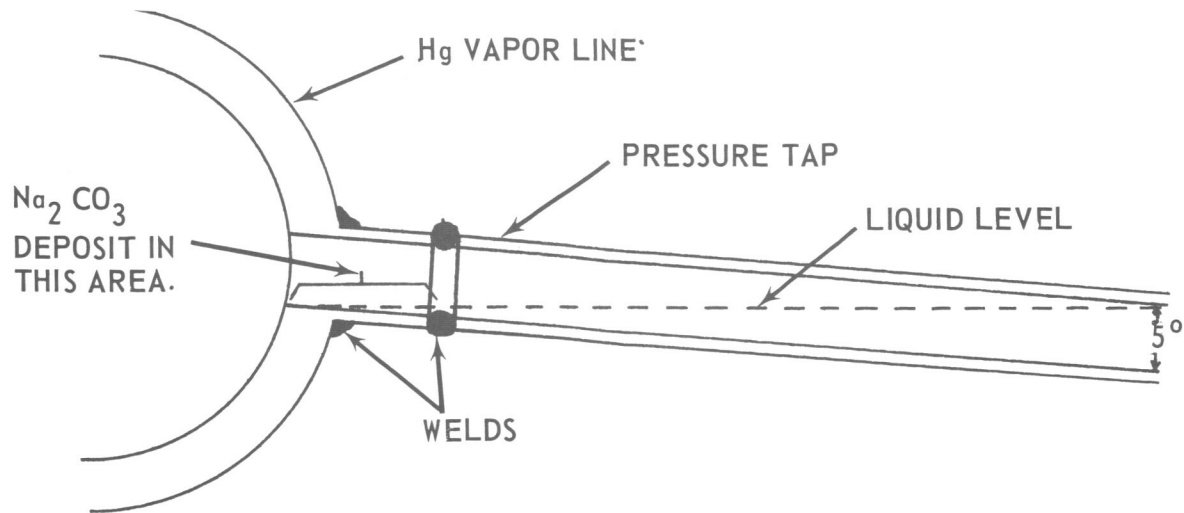
Total length (in.)	9.5
Internal diameter (in.) x length (in.), each region	
Region 1 (inlet)	0.147 x 4.0
Region 2 (middle)	0.104 x 3.0
Region 3 (exit)	0.074 x 2.5
Calming length (in.) and L/D ratio each section	
Region 1	3.0, 20.4
Region 2	2.0, 19.2
Region 3	1.5, 20.3
9Cr-1Mo sample length (identical all regions) (in.)	0.5

Performance

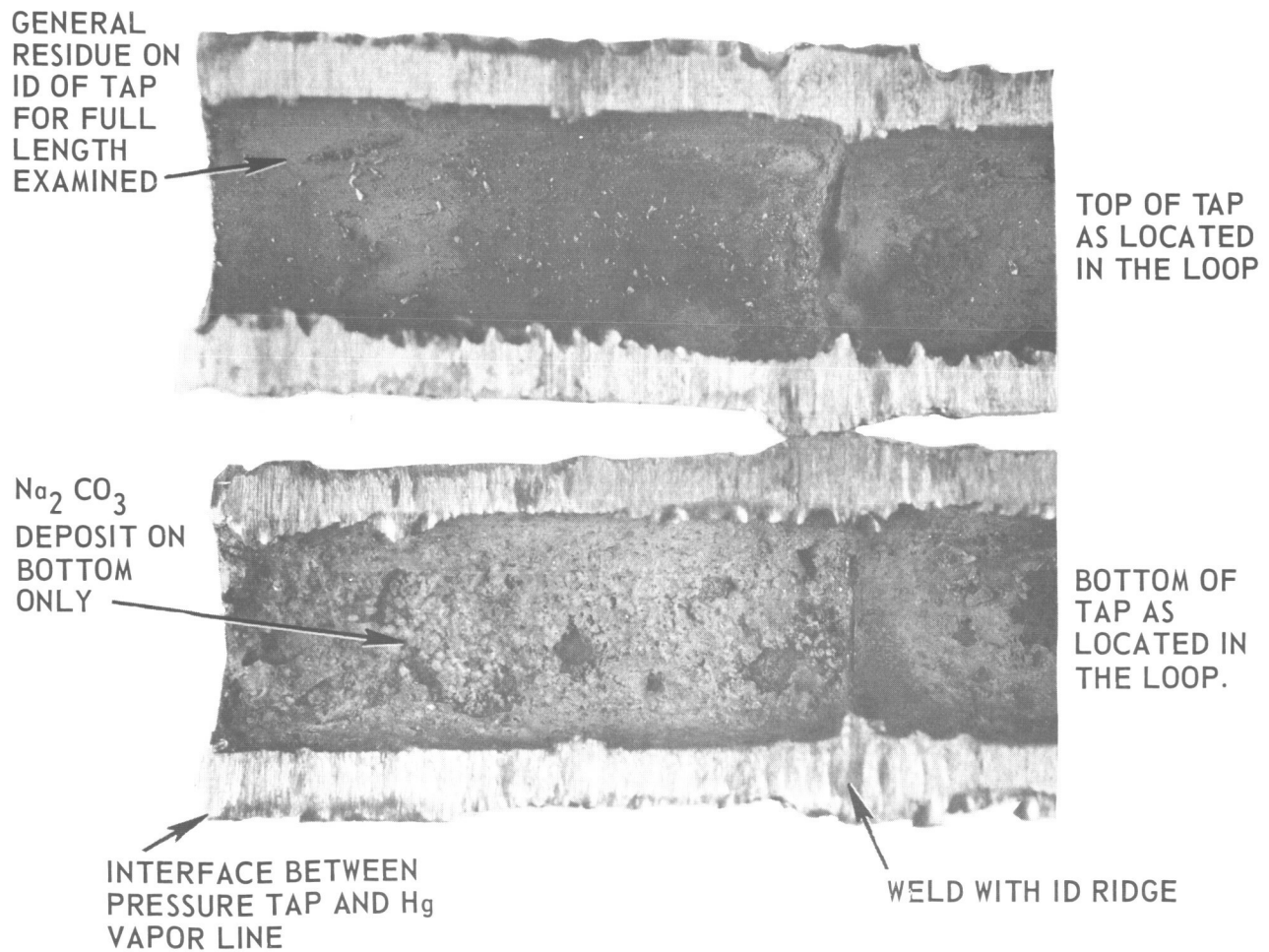
Hg flow rate (lb/hr)	729.8
Hg pressure drop (psi)	14.2
Hg inlet temperature (°F)	1100
Wall recession in 200 hr (mils)	
Region 1	3.07
Region 2	5.40
Region 3	8.68
Initial Hg velocity (fps) and Reynolds number*	
Region 1	2.25, 40500
Region 2	4.5, 57200
Region 3	9.0, 80900

* Note: Fluid properties are evaluated at 800°F.

866-NF-1147/A



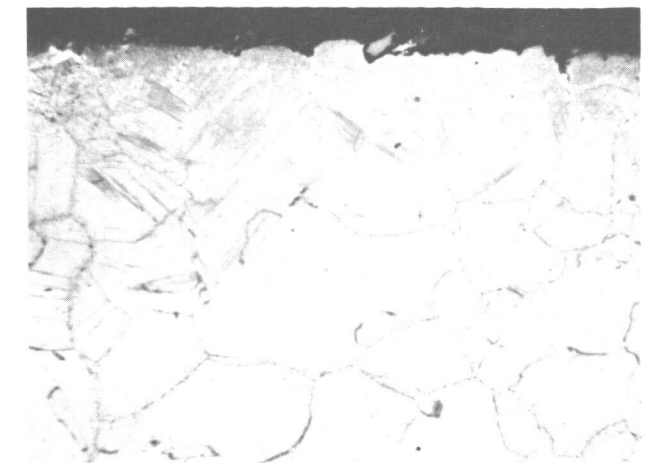
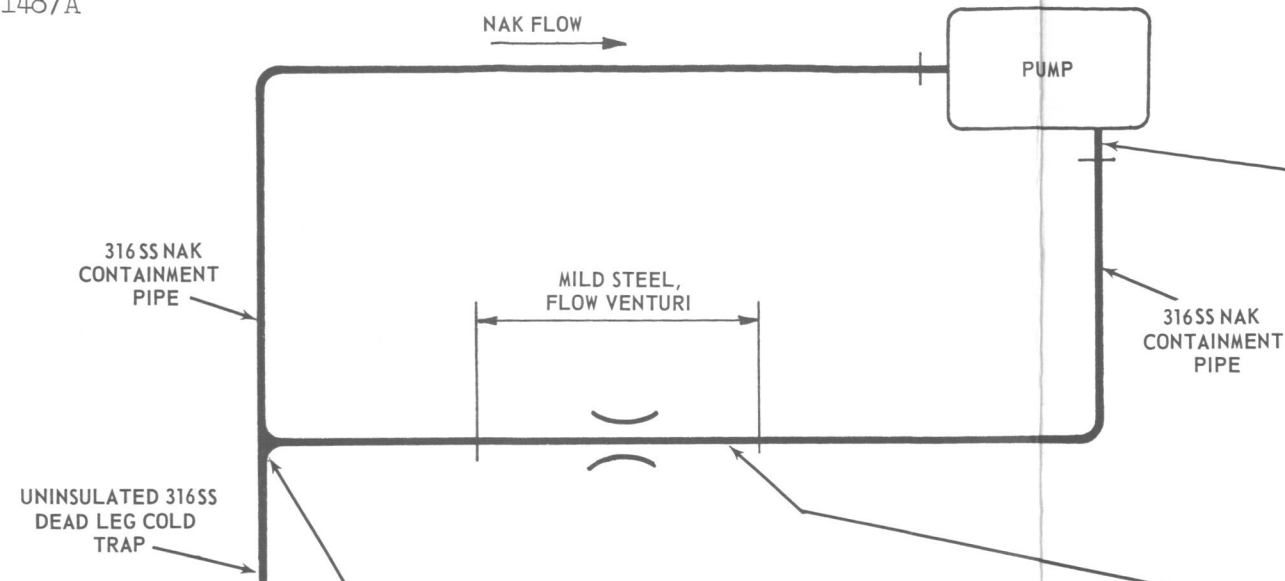
A. SCHEMATIC OF PRESSURE TAP INSTALLATION CONFIGURATION



B. LONGITUDINAL TAP SECTION. APPROXIMATELY 5X.

Typical Appearance of ID of Pressure Tap from
Hg Vapor Line of PCS-1 Phase IV After Step 2 Operation

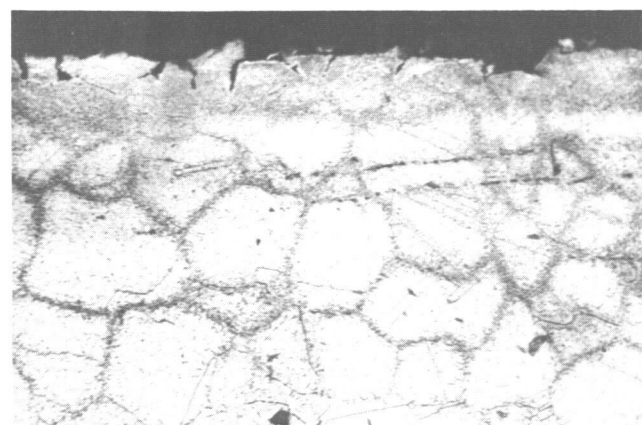
866-NF-1148/A



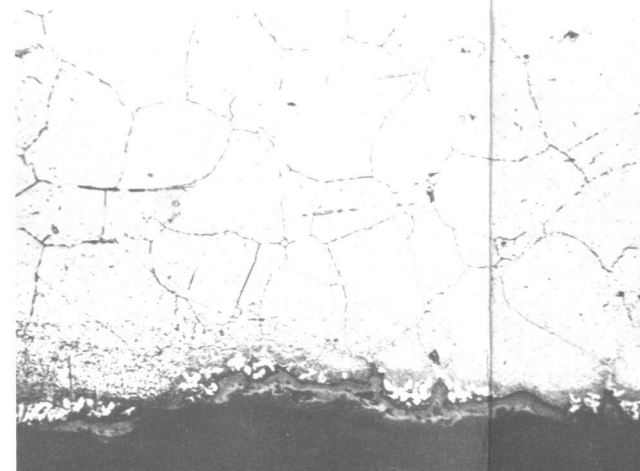
L-9474 INSIDE SURFACE CARBURIZATION

500 X

304SS PUMP DI



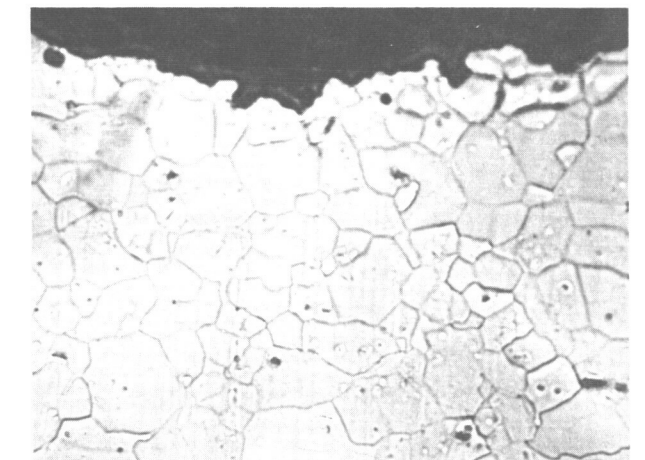
L-9471 INSIDE SURFACE CARBURIZATION 500X



L-9473 OUTSIDE SURFACE OXIDATION

316SS PIPE

ETCHANT: 10% OXALIC ACID ELECTROLYTIC

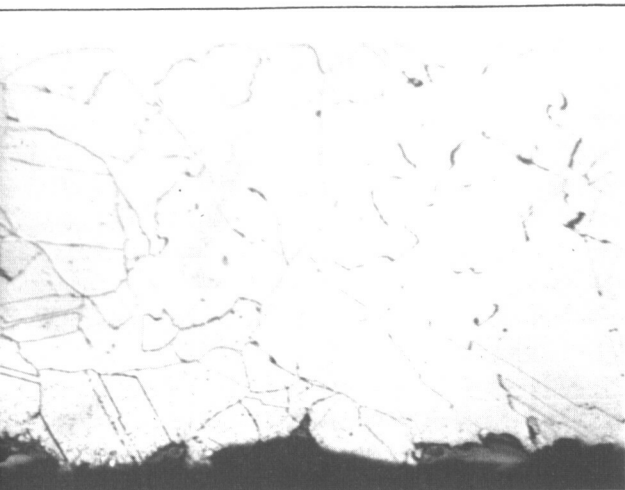


L-9476 INSIDE SURFACE DECARBURIZATION 500X

MILD STEEL PI

Microstructure of Specimens Removed from LNL-3 After 3130 hours of Isothermal System Operation at Between 1125 and 1175°F

Figure 2 -1

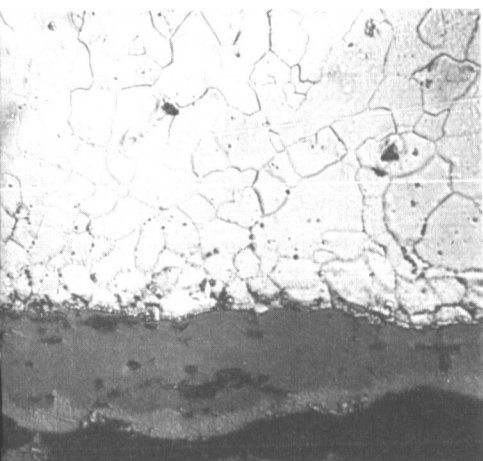


-9475

OUTSIDE SURFACE OXIDATION

CHARGE

ETCHANT: 10% OXALIC ACID ELECTROLYTIC



← SURFACE
OXIDE

OUTSIDE SURFACE OXIDATION

ETCHANT: PICRAL

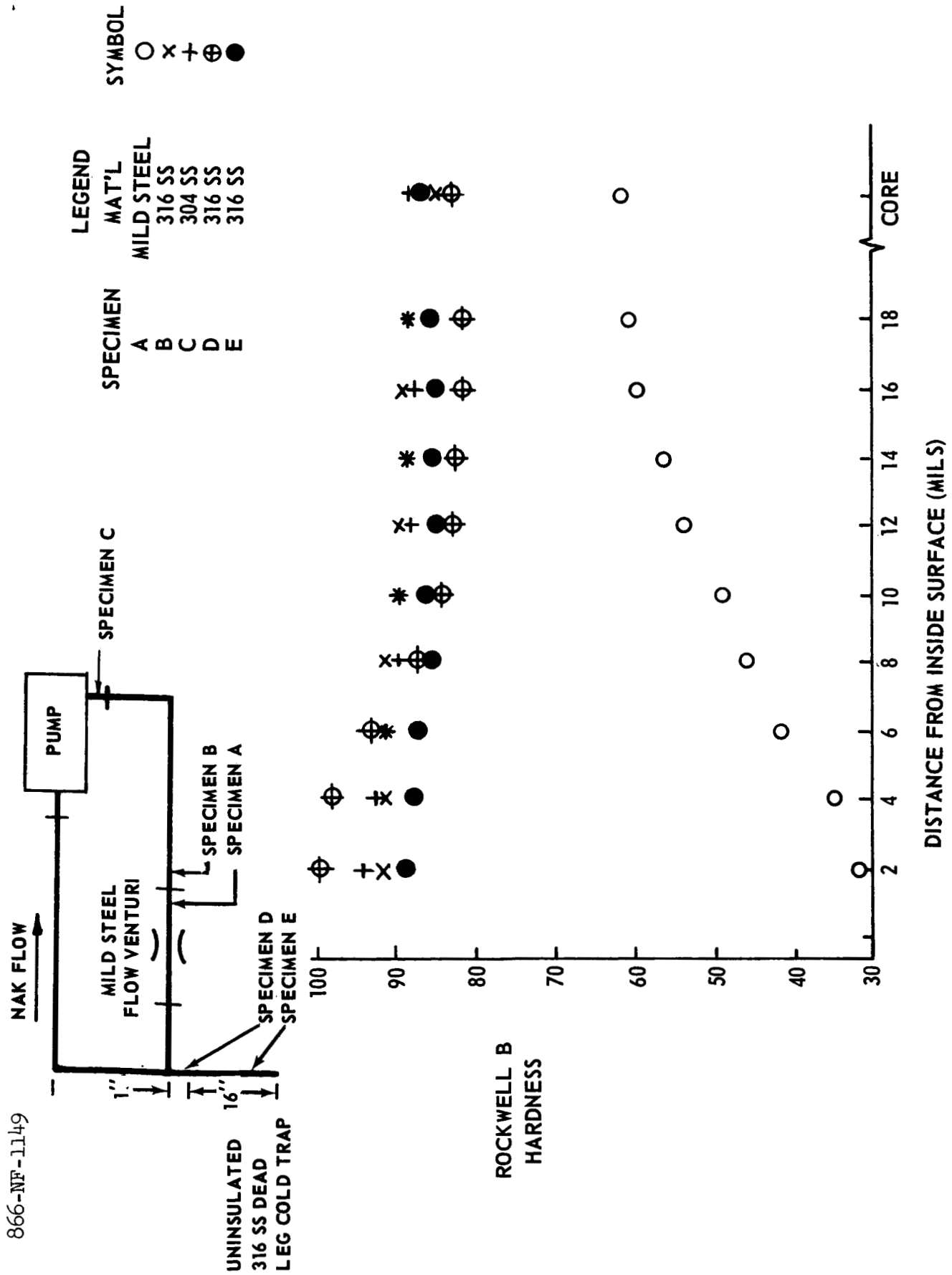
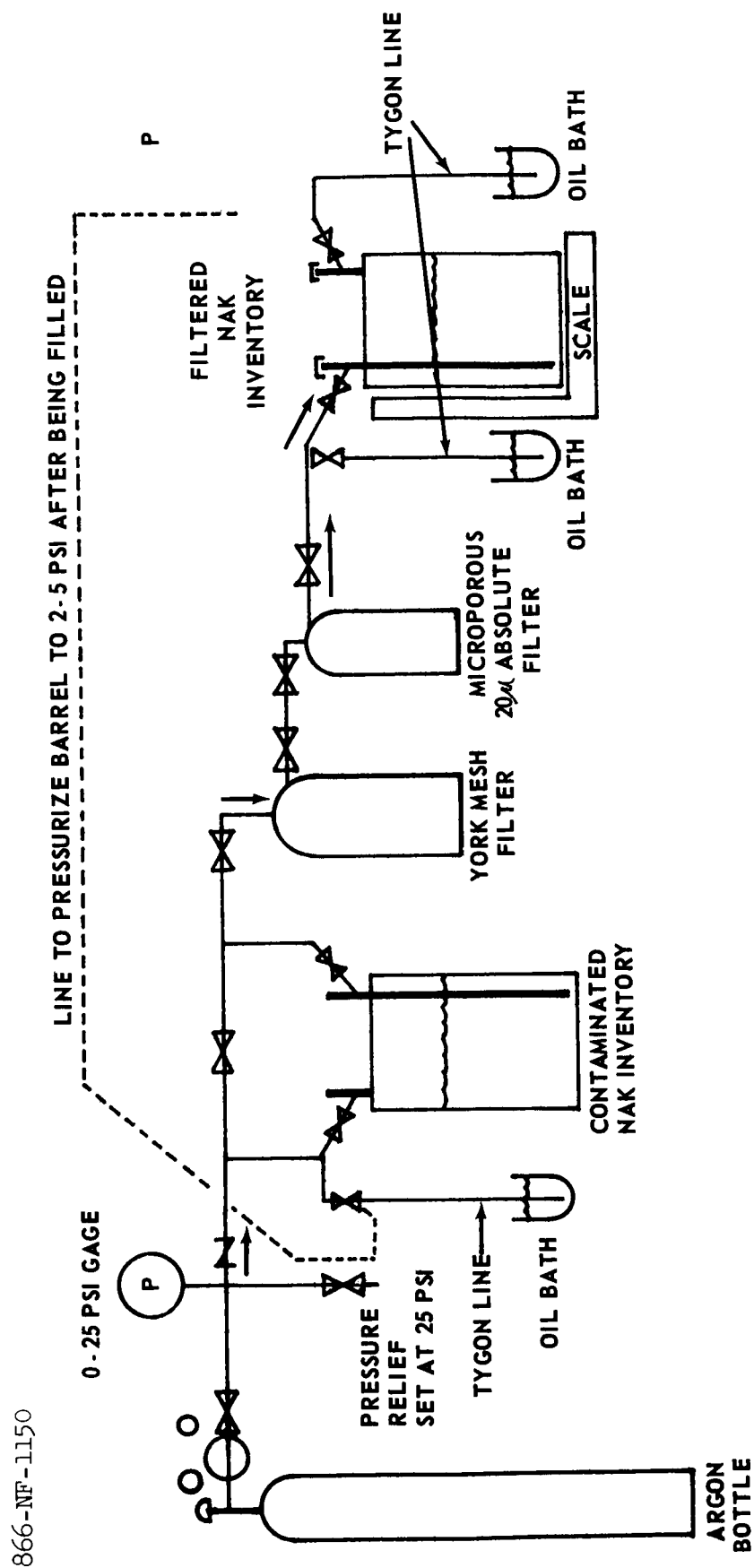


Figure 3

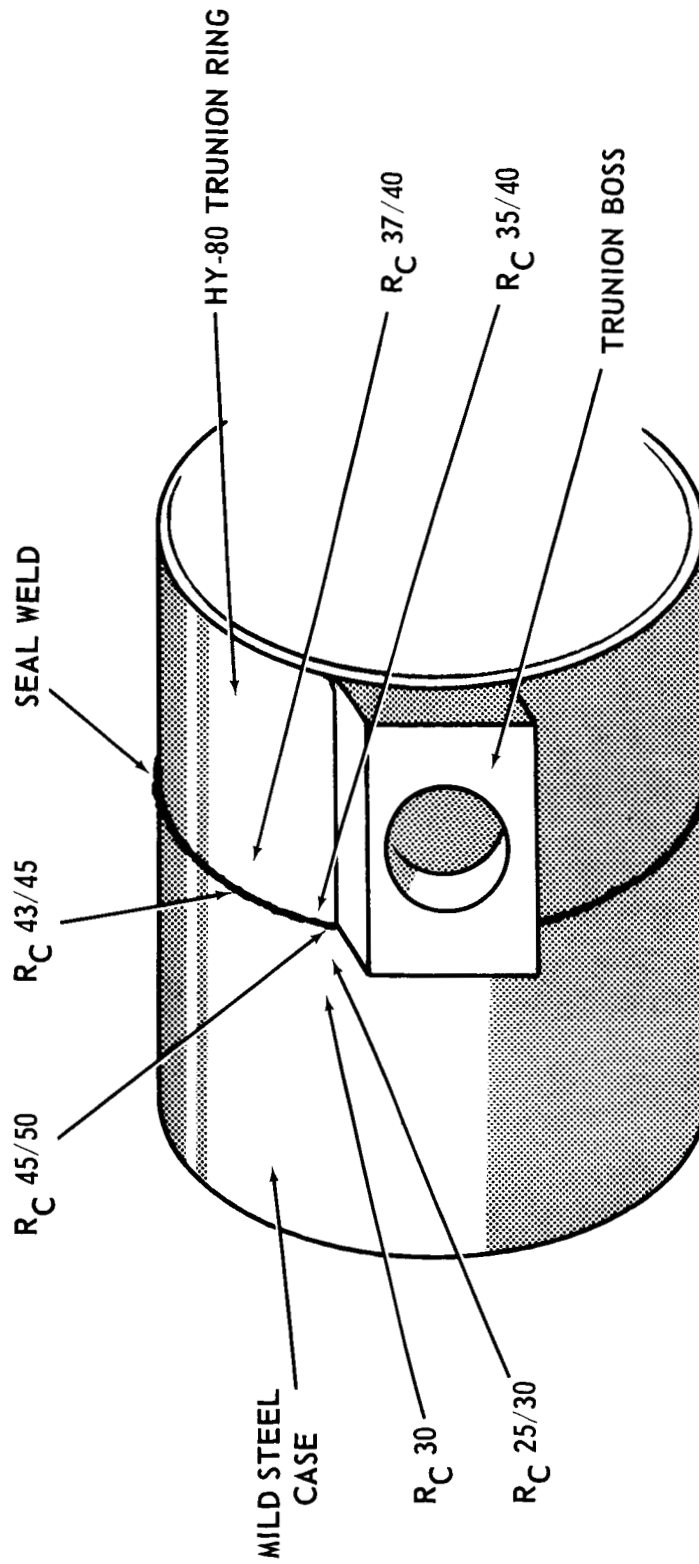
Rockwell Hardness Traverse from Inside Pipe Surface of INL-3 Pipe Specimens Removed After 3130 hours of Isothermal System Operation at Between 1125 and 1175°F (Hardness Converted From Knoop Microhardness Reading Taken with a 100-mg Load)



Schematic of System for NaK Purification by Filtration

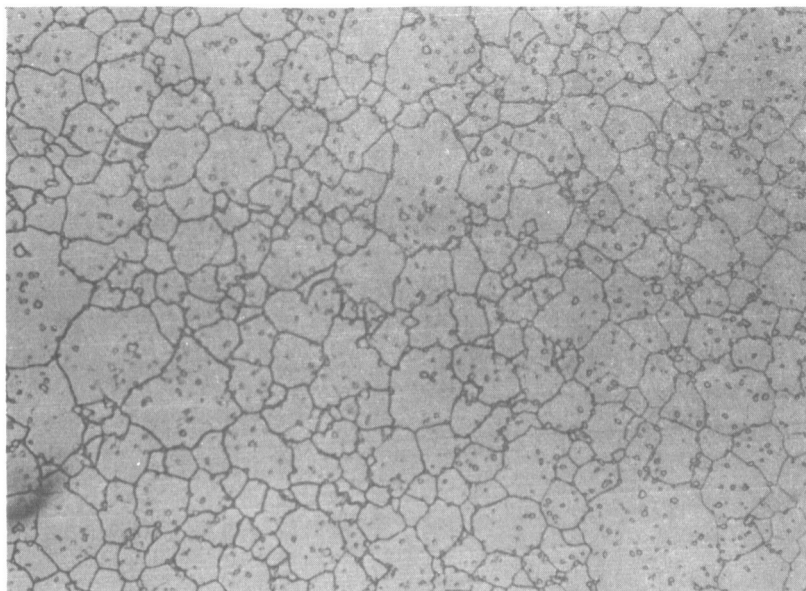
Figure 4

866-NF-1151



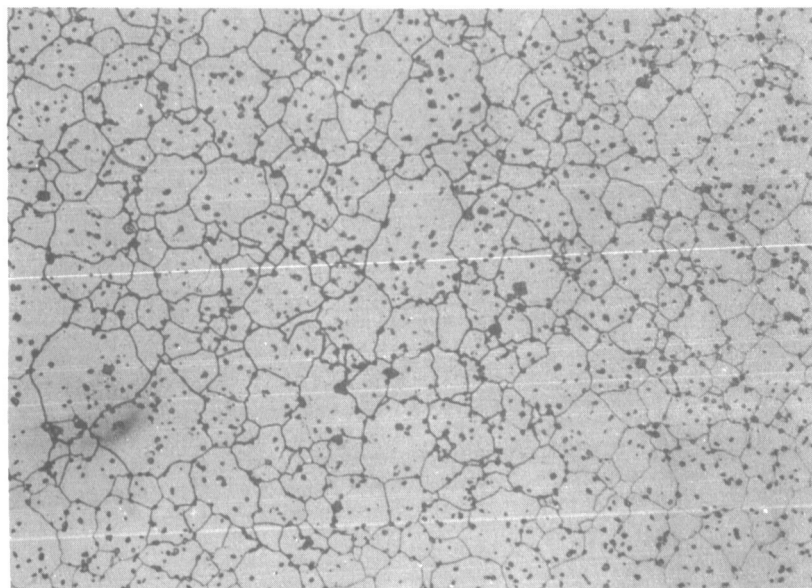
Qualitative Hardness Test Results on SNAP-8 Alternator (S/N 481489)
 After 140 hours of Operation in PCS-1 Phase IV Step 2
 (Higher Numerical Values Indicate Greater Hardness)

Figure 5



L-9428

250X



L-9429

STAINED WITH MURAKAMI 'S
REAGENT AFTER ETCHING

250X

S-816 Cobalt-Base Alloy After an Exposure of 2700 hours at 1200°F.

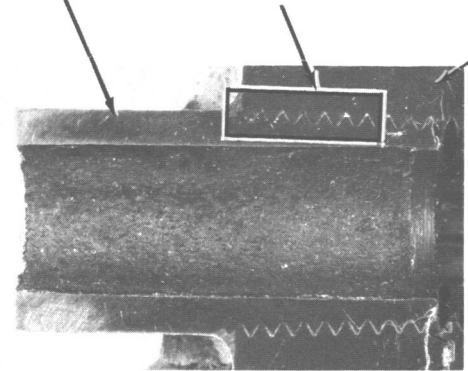
In the Bottom Photomicrograph the Darkened Islands are Carbides and the Small Undarkened Islands may be Sigma Phase

Figure 6

MILD STEEL FITTING

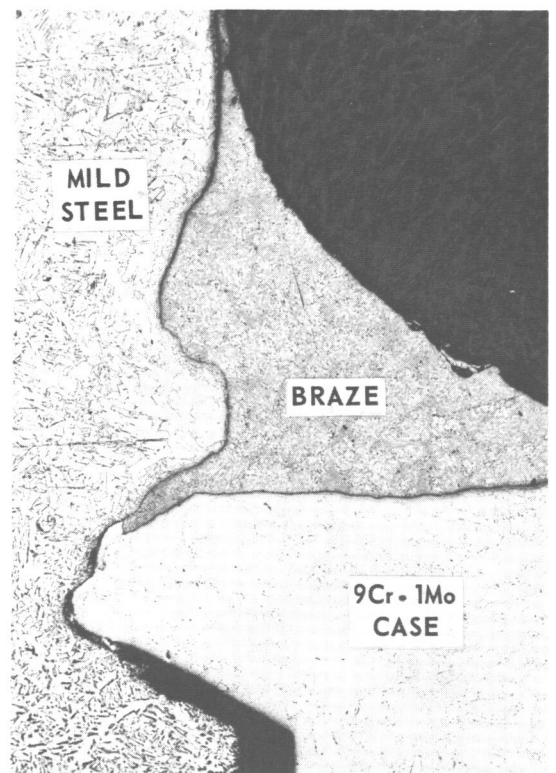
SEE ENLARGED
AREA AT RIGHT

9Cr-



L-9482

2X



L-9348

50 X

B. UNEXPOSED BRAZED

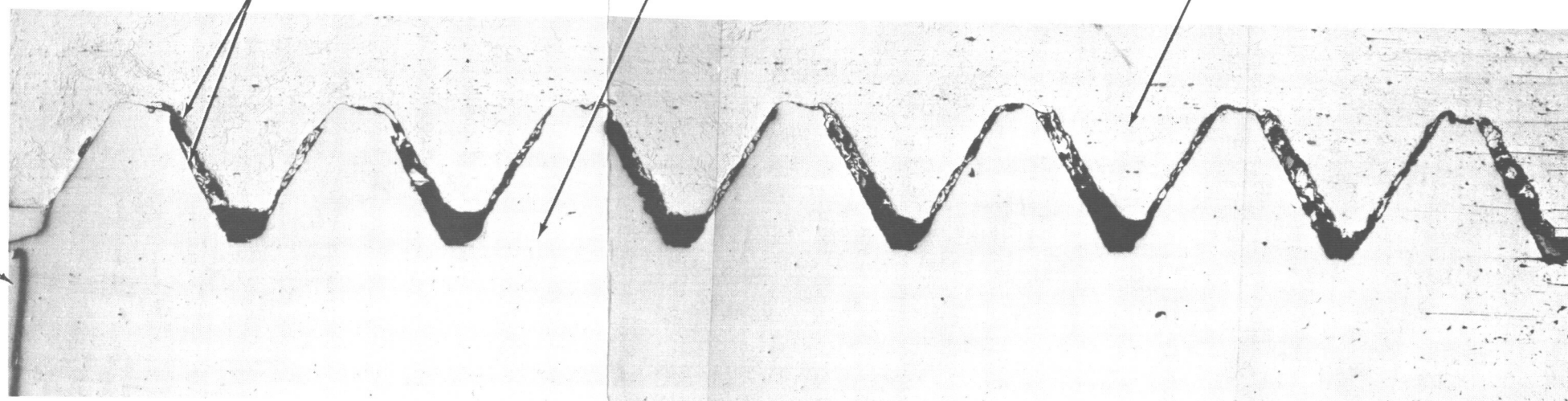
IMo TA CASE

EASY FLO NO. 3
BRAZE JOINT

DRYSEAL PIPE THREAD

MILD STEEL FITTING

TA CASE

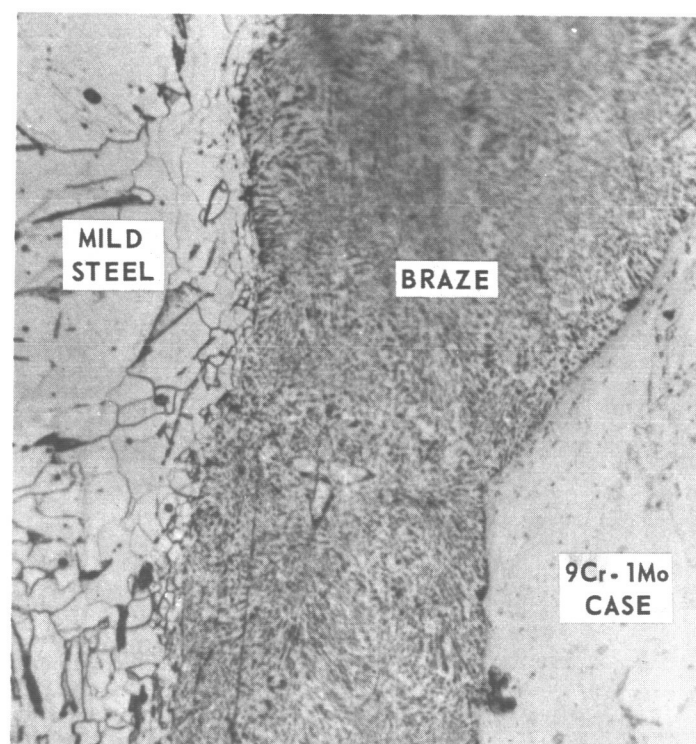


AS POLISHED

L-9501-3

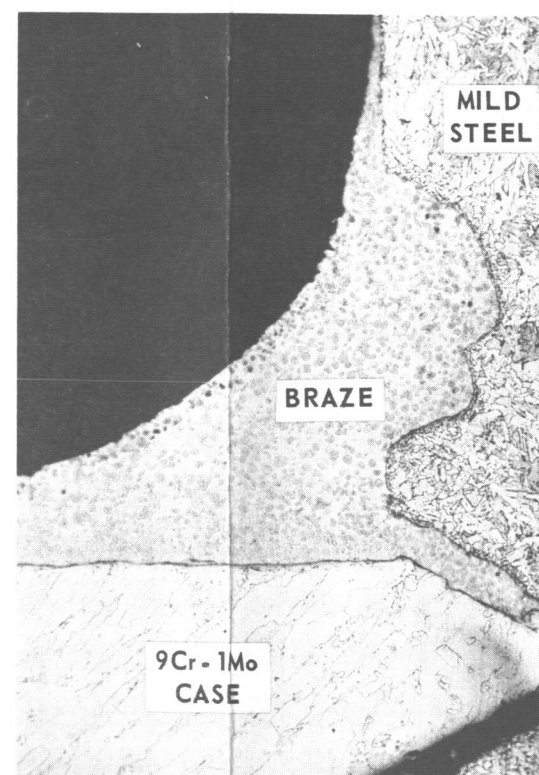
40 X

A. AS-BRAZED JOINT SHOWING JOINT CONTOUR AND ENGAGEMENT OF DRYSEAL PIPE JOINT BETWEEN TA CASE AND FITTING



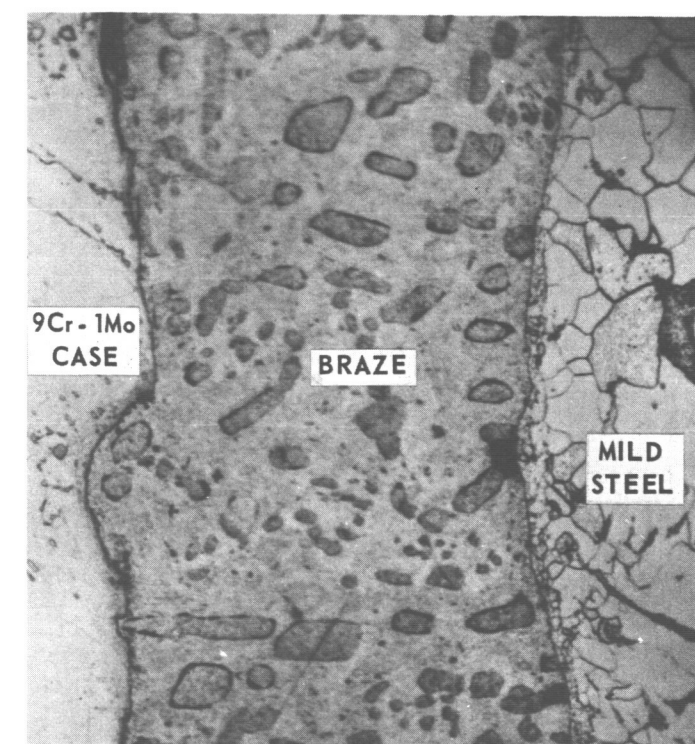
L-9494

500 X



L-9493

50 X



L-9495

500 X

JOINT. ETCHANT: VILLELA'S

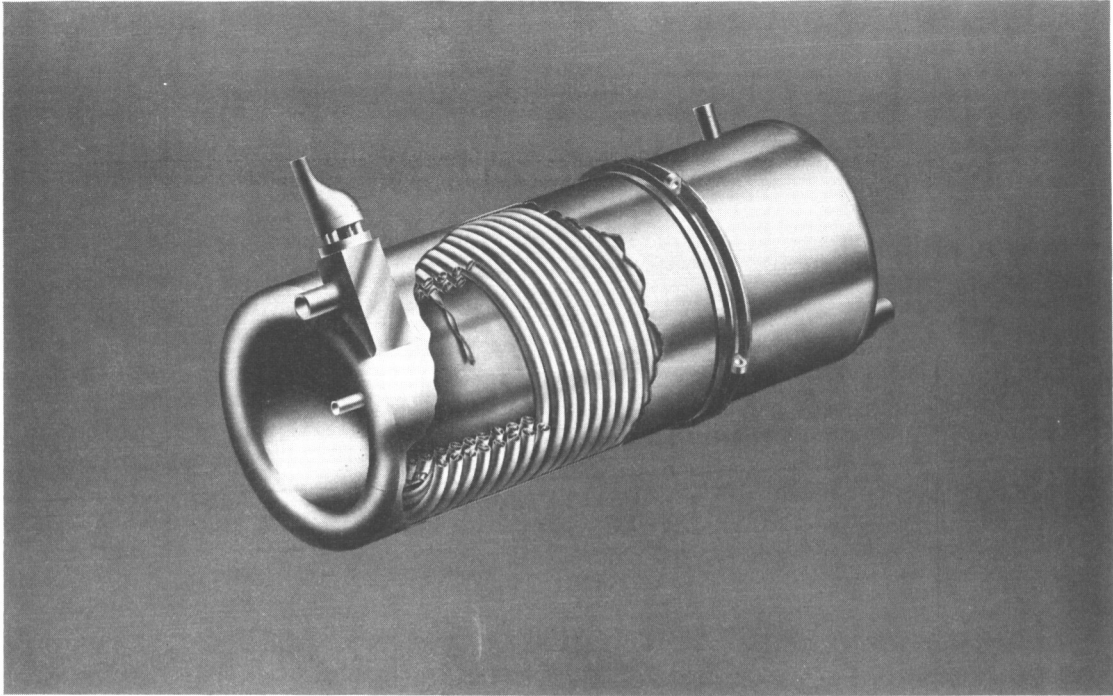
C. BRAZED JOINT AFTER EXPOSURE. NO CROSS-BOND DIFFUSION OCCURRED BUT BRAZE ALLOY HOMOGENIZED STRUCTURE IS SEEN. ETCHANT: VILLELA'S

7-2

Simulated TA Interstage Pressure Tap Braze Joint After 137 Hours at 1000°F in Air

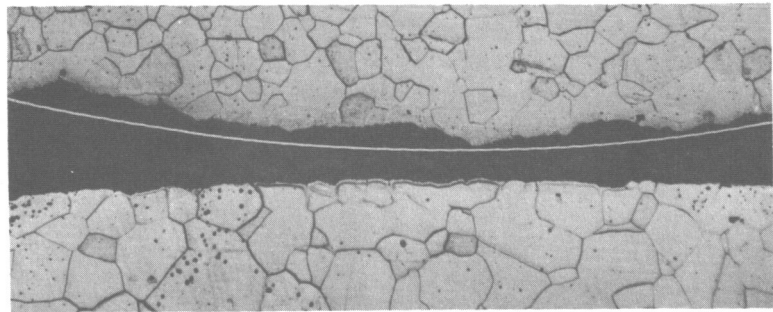
Figure 7-3

1065-568



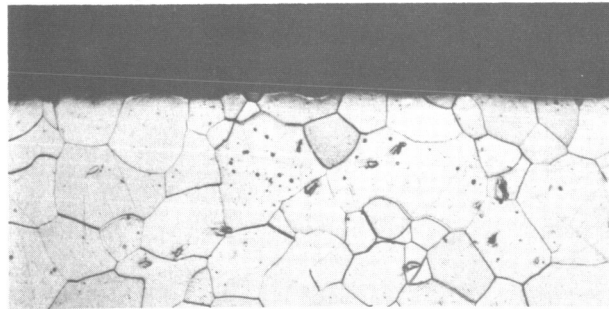
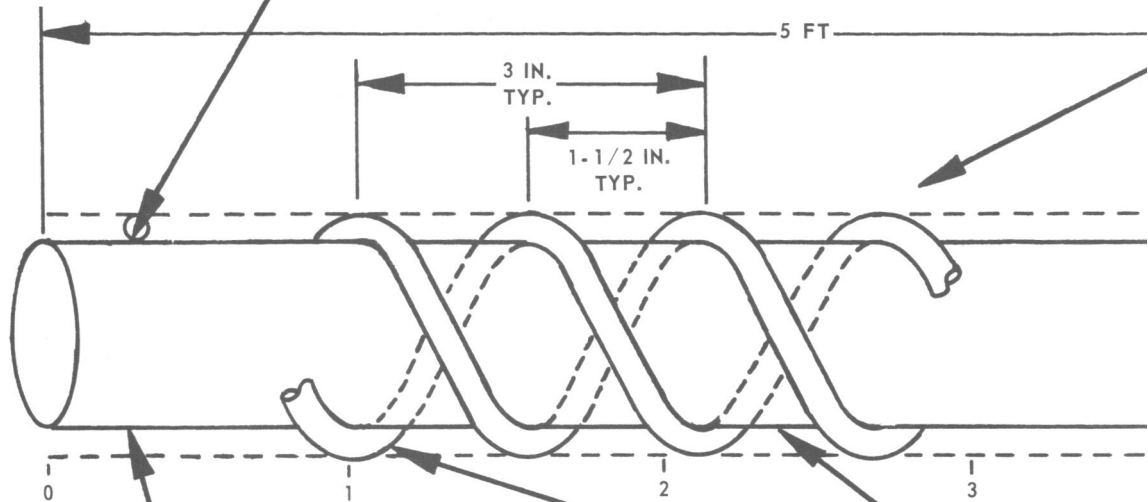
SNAP-8 Tube-in-Shell Boiler

Figure 8



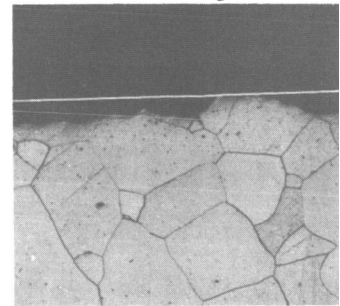
L-9103

WIRE-BAR (AT BOTTOM) INTERFACE

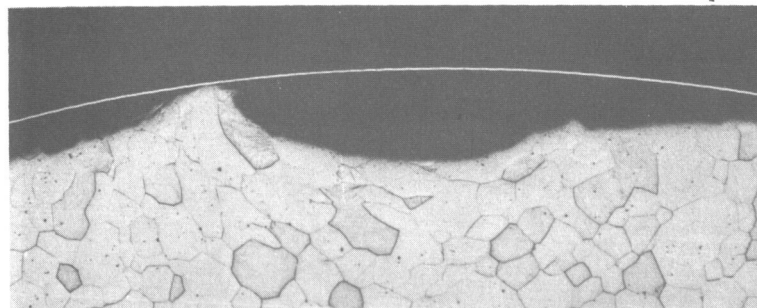


L-9101

BAR



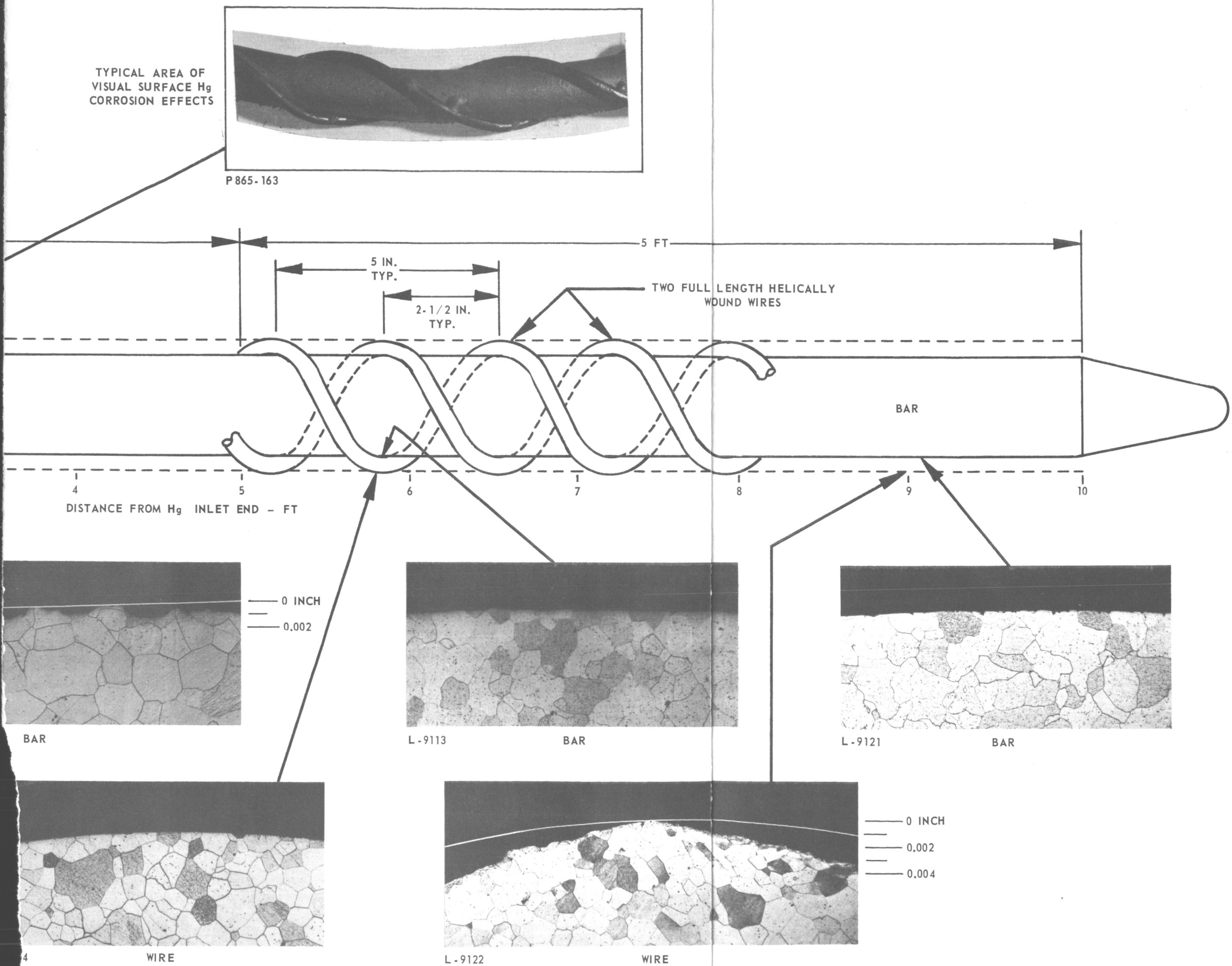
L-9111



L-9101

WIRE

0 INCH
0.002
0.004



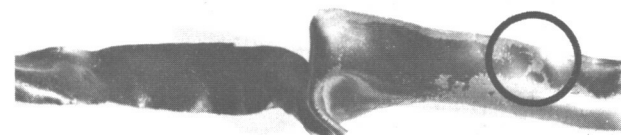
INDICATED WHITE LINE REPRESENTS PRESUMED ORIGINAL WIRE SURFACE. ETCHANT: VILLELA'S

9-2

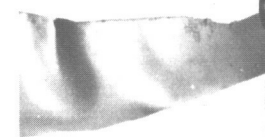
Hg Corrosion Effects on Core and Wire of Hg Inlet Flow Restriction Plug which Produced a Hg Velocity of 0.25 fps - Plug Removed from Tube Coil 0-4 of -1 Boiler After 1415 hours of Operation in RPL-2

9-3
Figure 1

B366-NF-1107/A

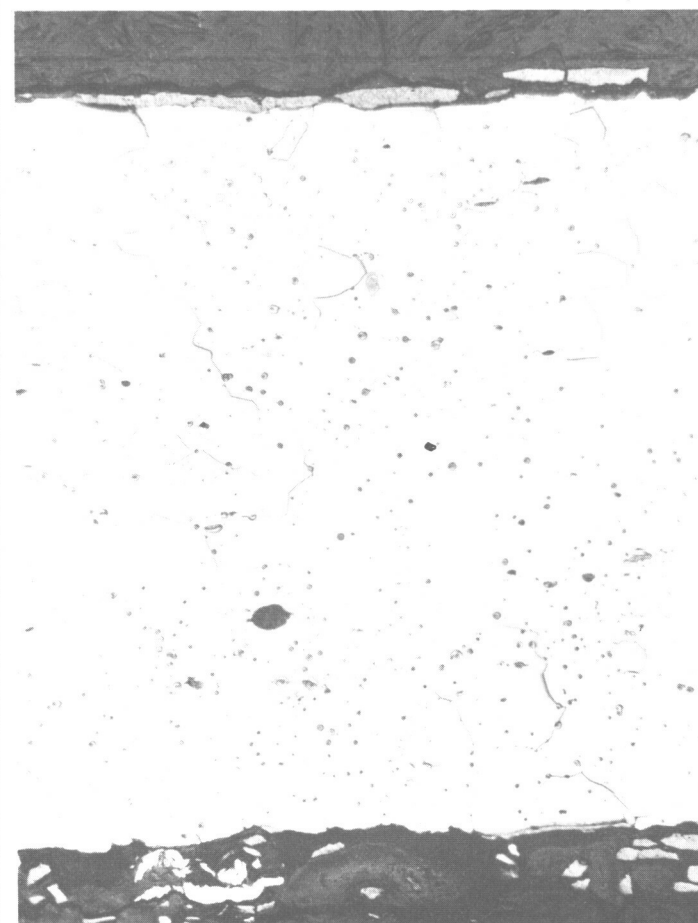


865-171



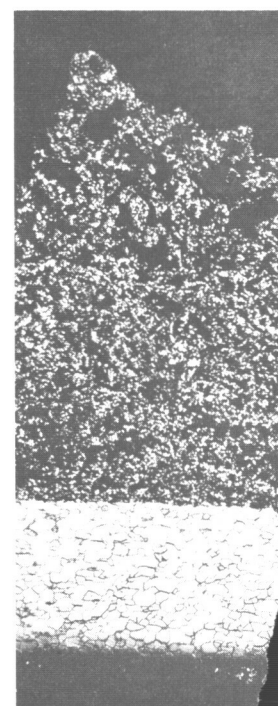
865-169

MACROGRAPHIC SECTIONS - IRREGULAR SH
CUTTING OF Hg TUBE



442

250 X



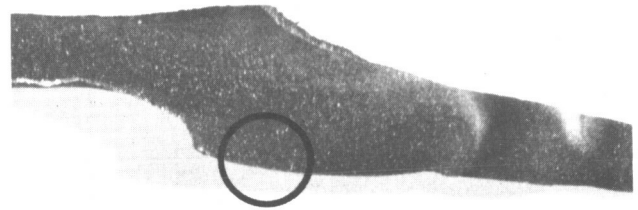
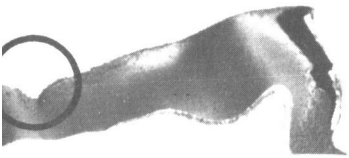
9145

MICROGRAPHIC SECTIONS (OF AREAS EN
0.016 INCH R

A. RIBBON AT 11 FT FROM Hg INLET
SHOWING ADHERENT SURFACE FILM

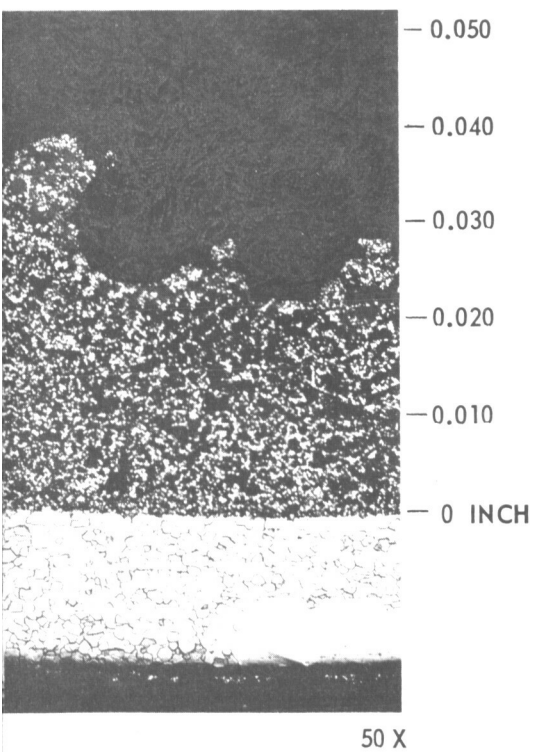
B. RIBBON
LET SHO
FER DEF
BY RIBB

Ribbon Turbulator from Var
After 14

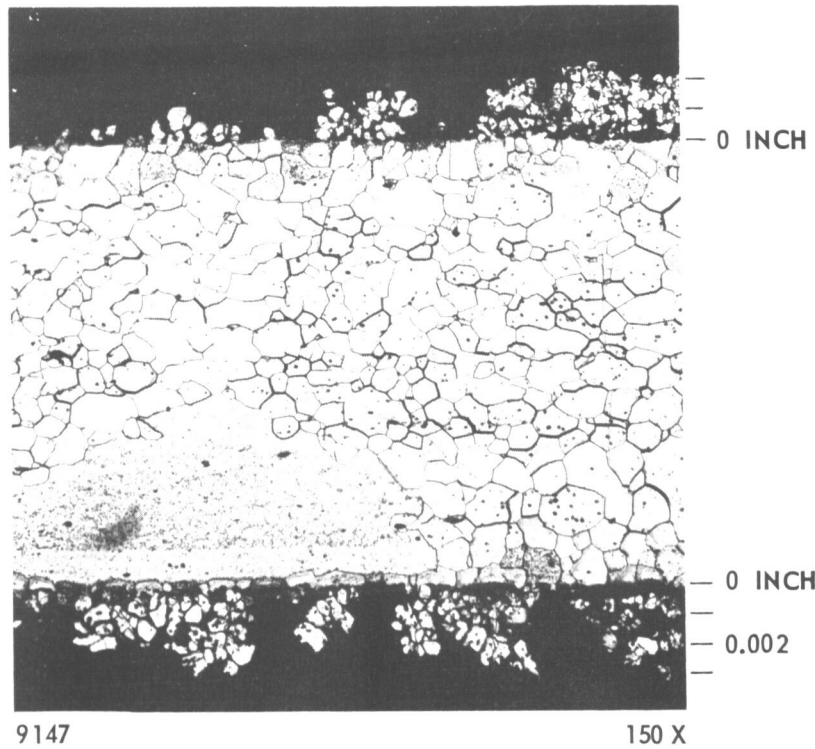


865-166

SHAPE OF RIBBON PRODUCED DURING LONGITUDINAL
CONTAINING INTERNAL RIBBON



50 X



150 X

(CIRCLED ABOVE) SHOWING BOTH SURFACES ACROSS
RIBBON THICKNESS

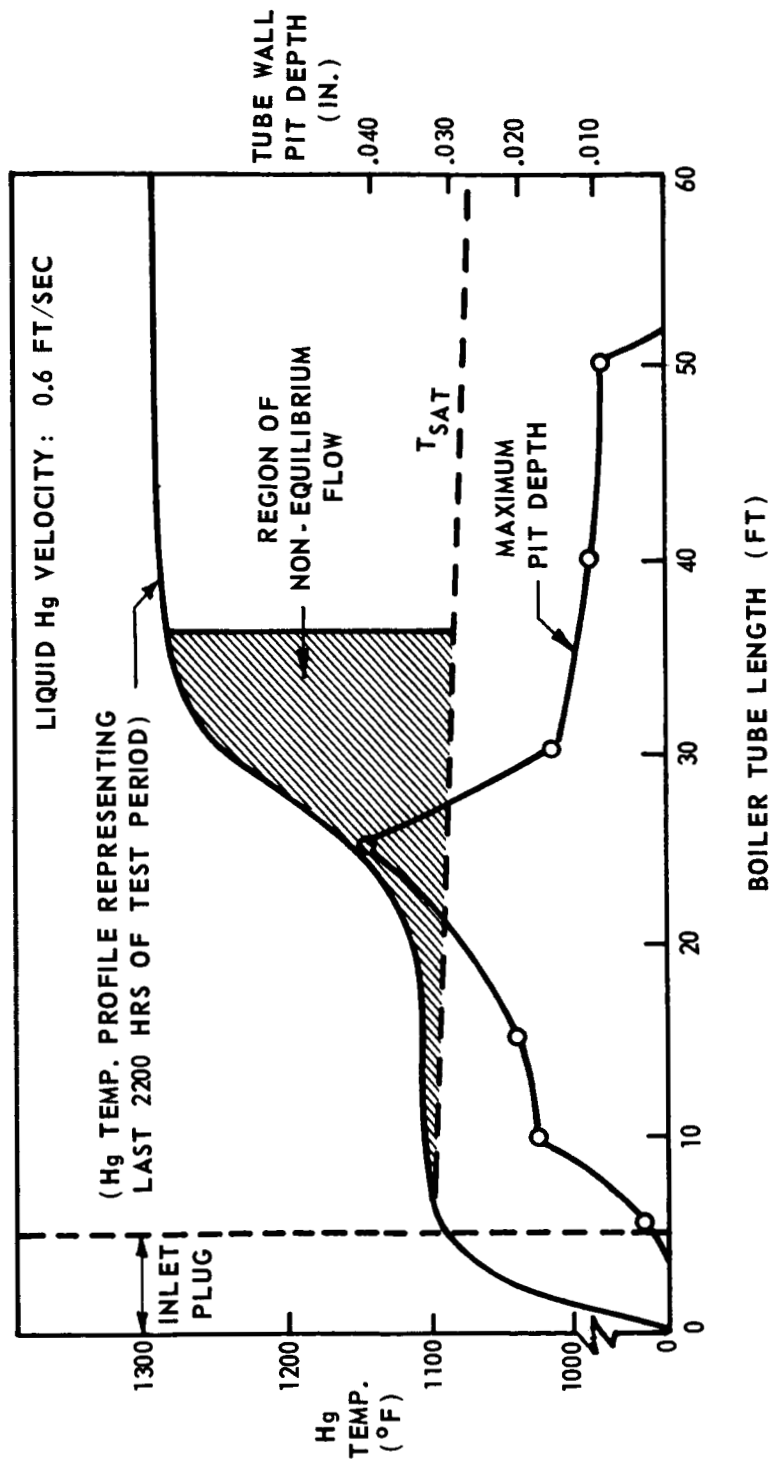
38-1/2 FT FROM Hg IN-
LET SHOWING SPONGY MASS TRANS-
FER DEPOSIT IN POCKETS FORMED
DURING DEFORMATION

C. RIBBON AT 46-1/2 FT FROM Hg IN-
LET SHOWING DENDRITIC UNIFORM
MASS TRANSFER DEPOSIT

Sections of Tube Coil 0-4 of -1 Boiler
after 100 hours of Operation in RPL-2

Figure 10-2

666-46



Corrosion Loop 3 1/19-Scale Boiler Test (Time: 4400 hours)

Figure 11

666-47

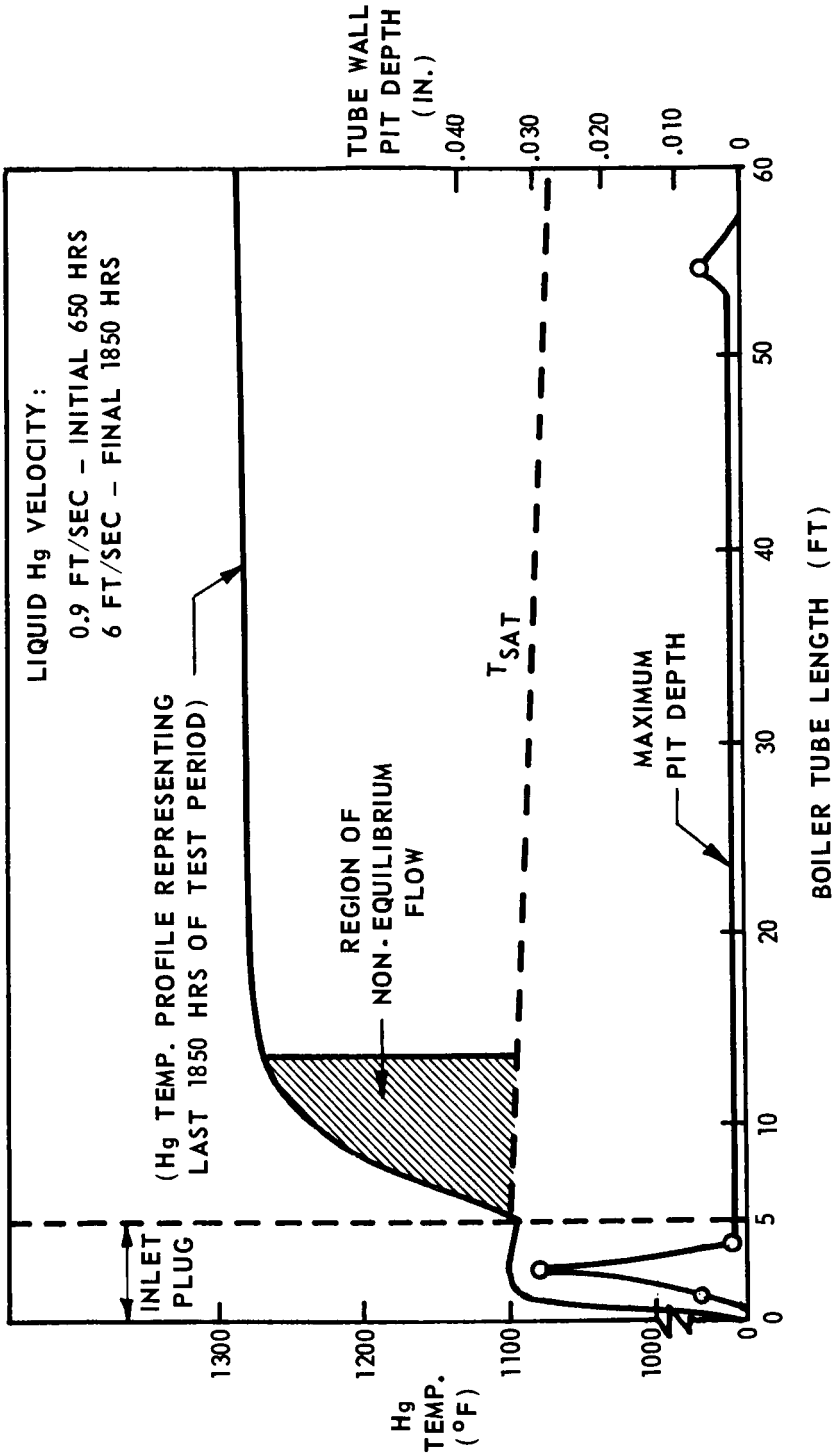


Figure 12

Corrosion Loop 4 1/19-Scale Boiler Test (Time: 2500 hours)

666-33

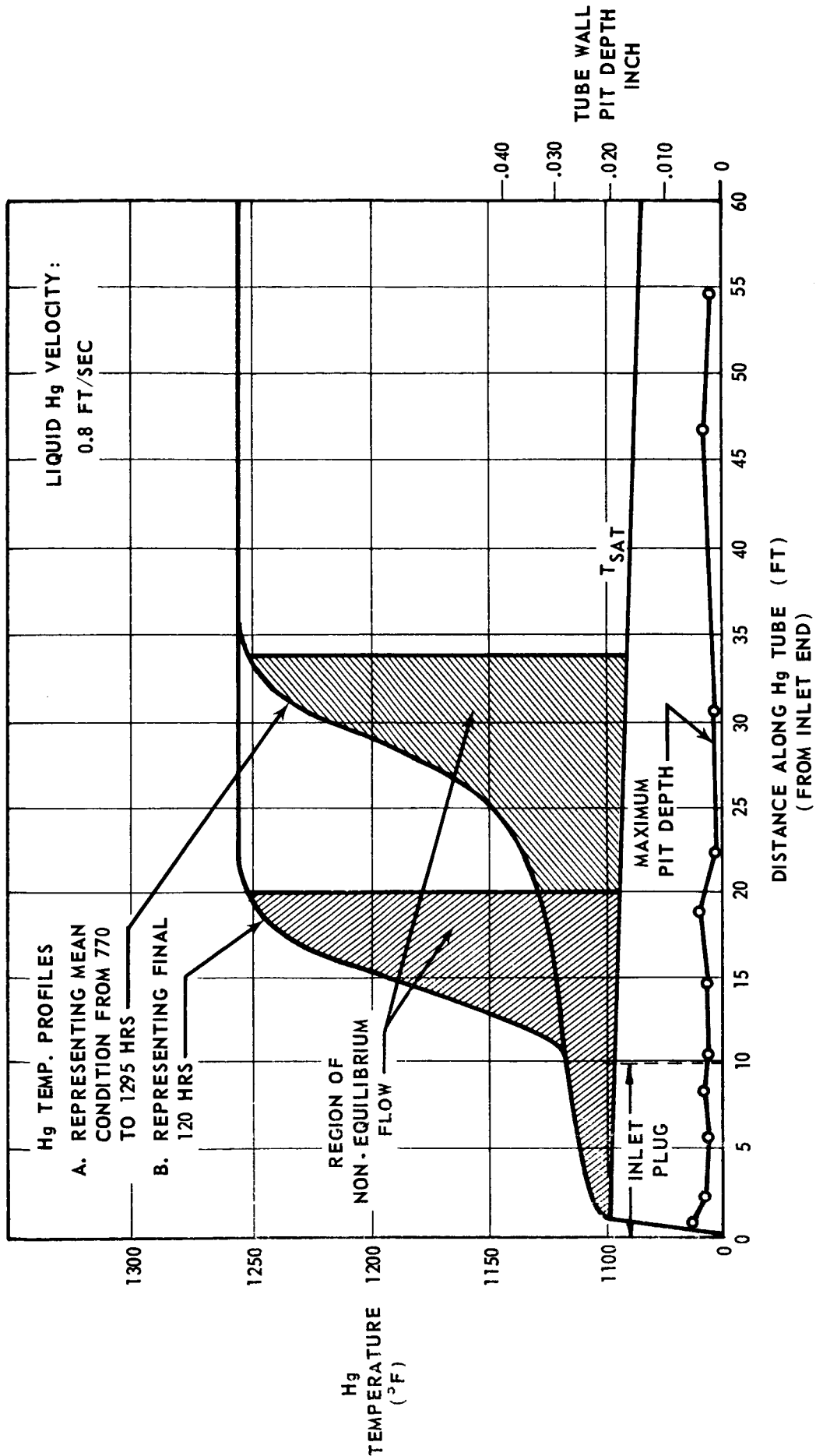


Figure 13

RPL-2 Full-Scale Tube-in-Shell Boiler Test (Time: 1425 hours)

866-NF-1122

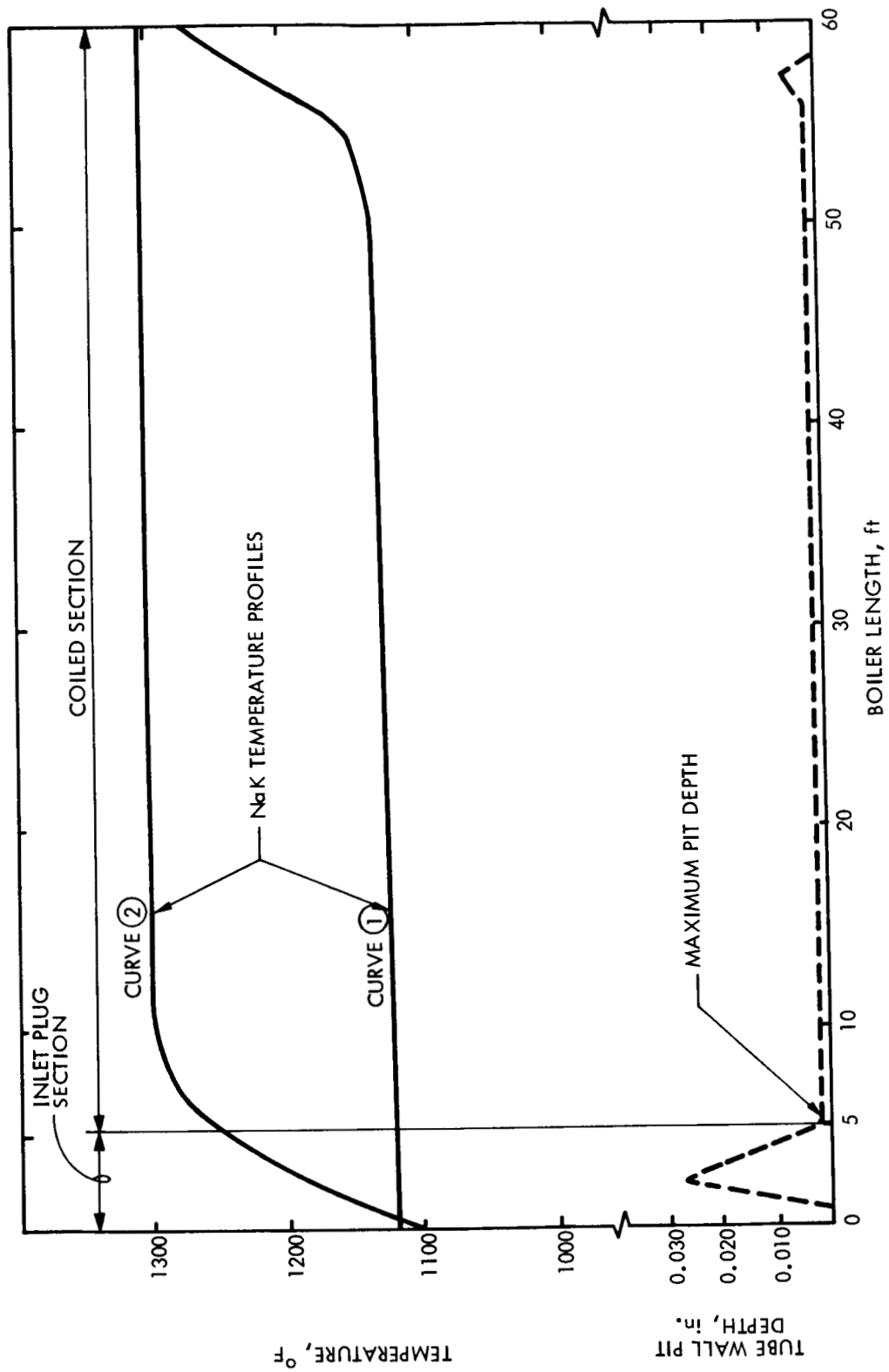
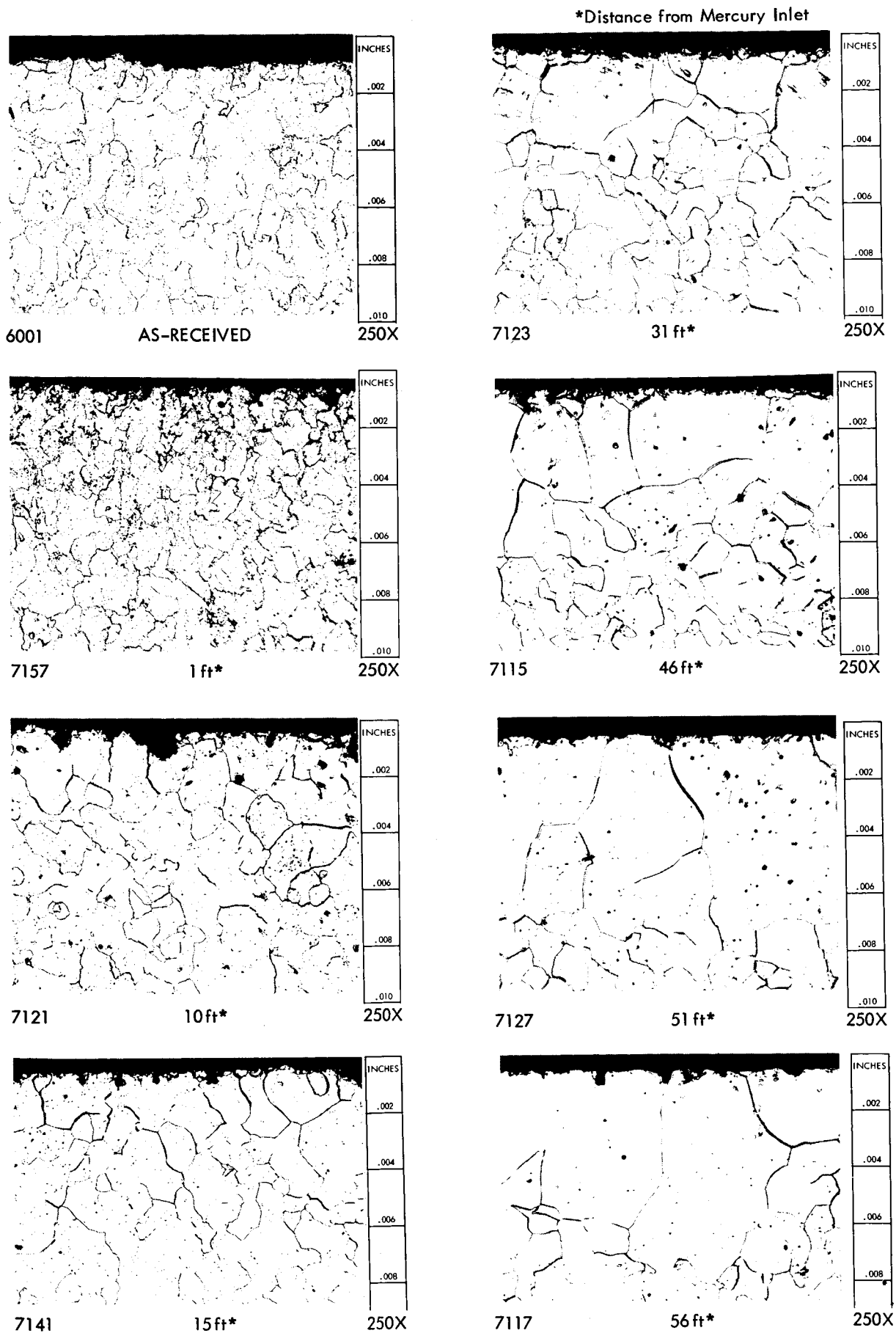
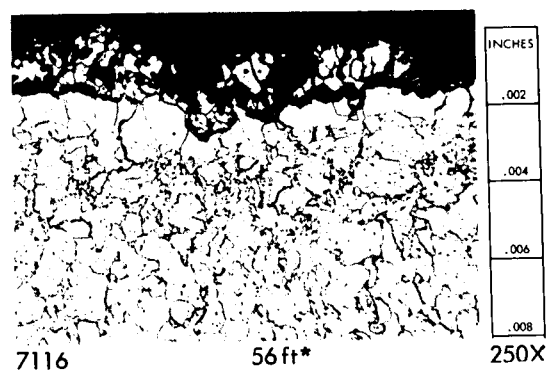
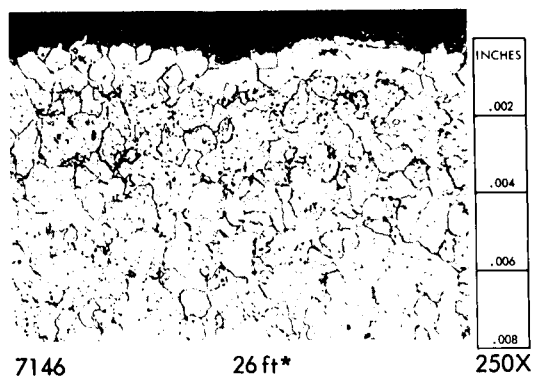
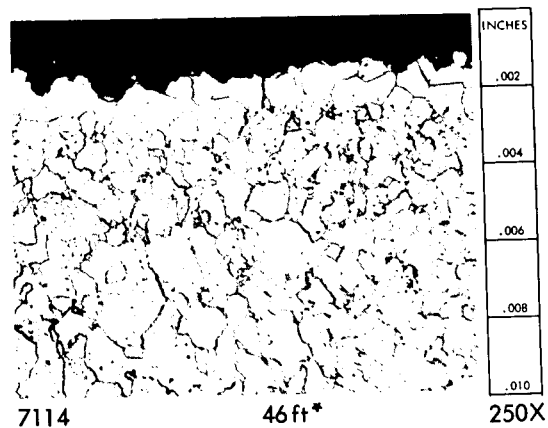
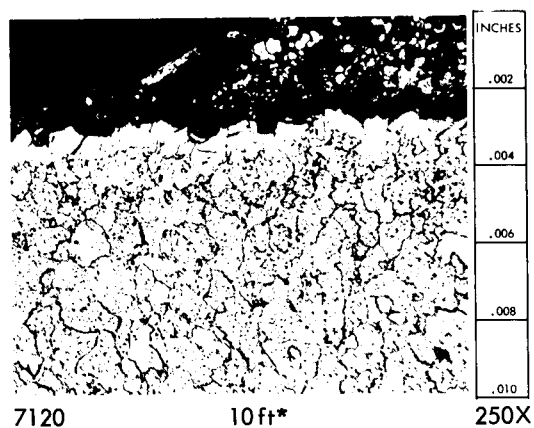
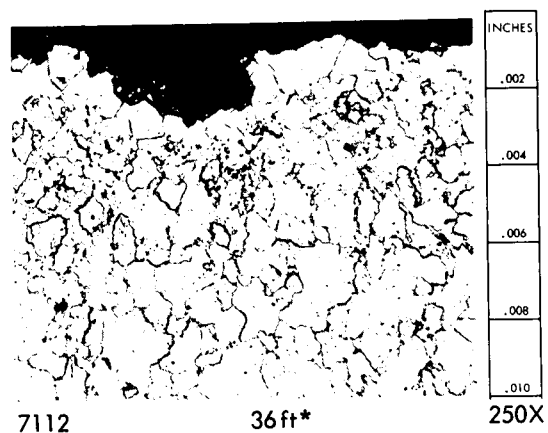
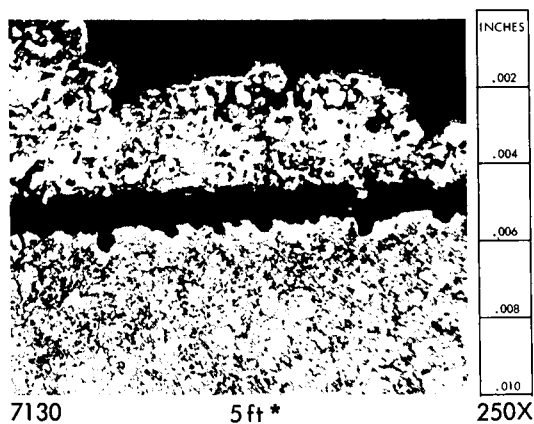
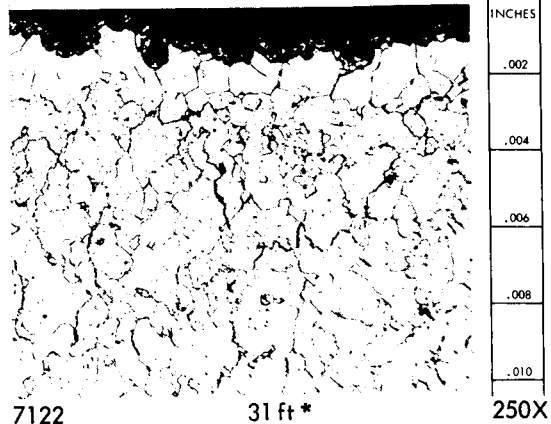
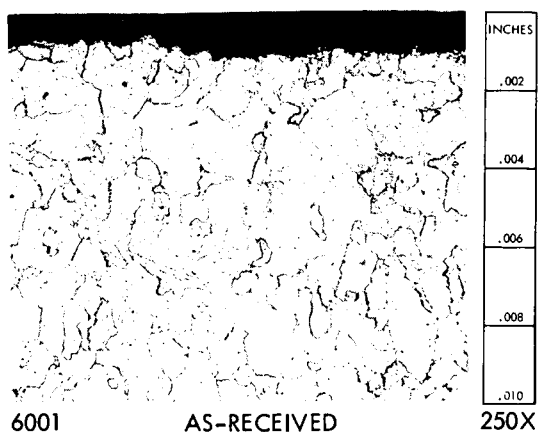


Figure 14

Typical NaK Temperature Profiles And Pit Depths CL-4 Boiler



MICROSTRUCTURE OF THE NaK SIDE OF THE 9Cr-1Mo TUBING - CL 4 BOILER



MICROSTRUCTURE OF THE MERCURY SIDE OF THE 9Cr-1Mo
TUBING - CL 4 BOILER COILED SECTION

Figure 16

866-NF-1125

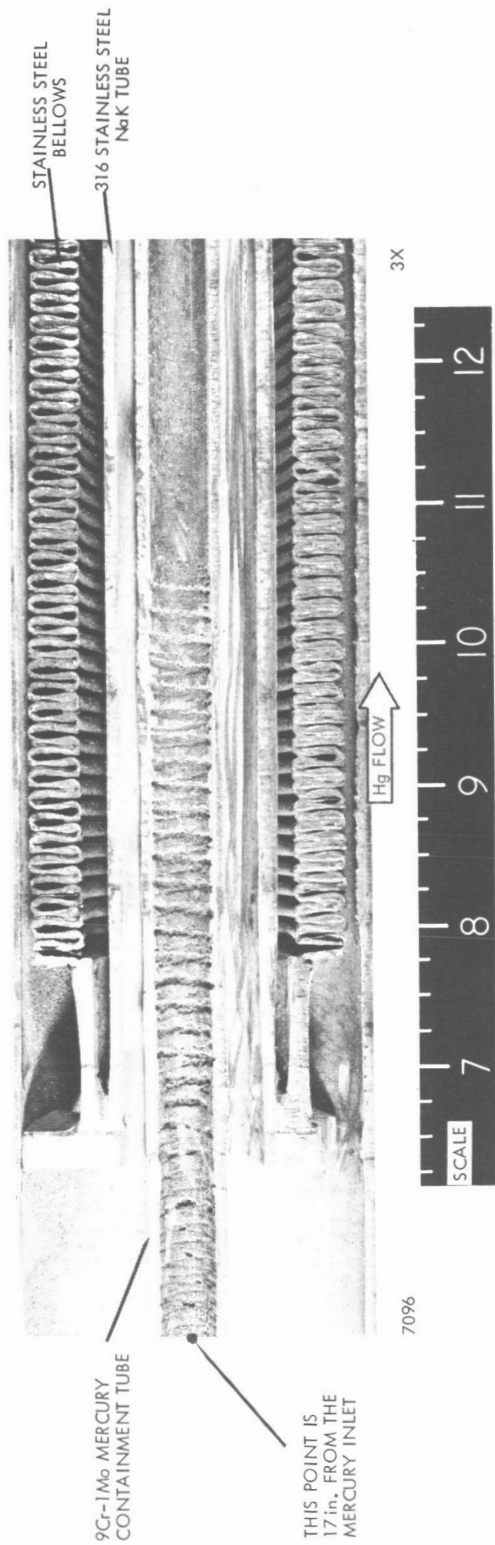


PHOTO MICROGRAPH - CL 4 BOILER INLET SECTION

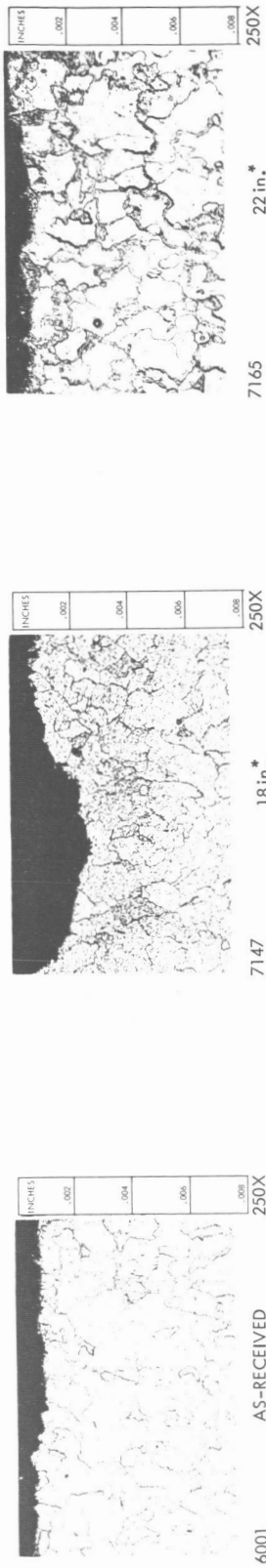


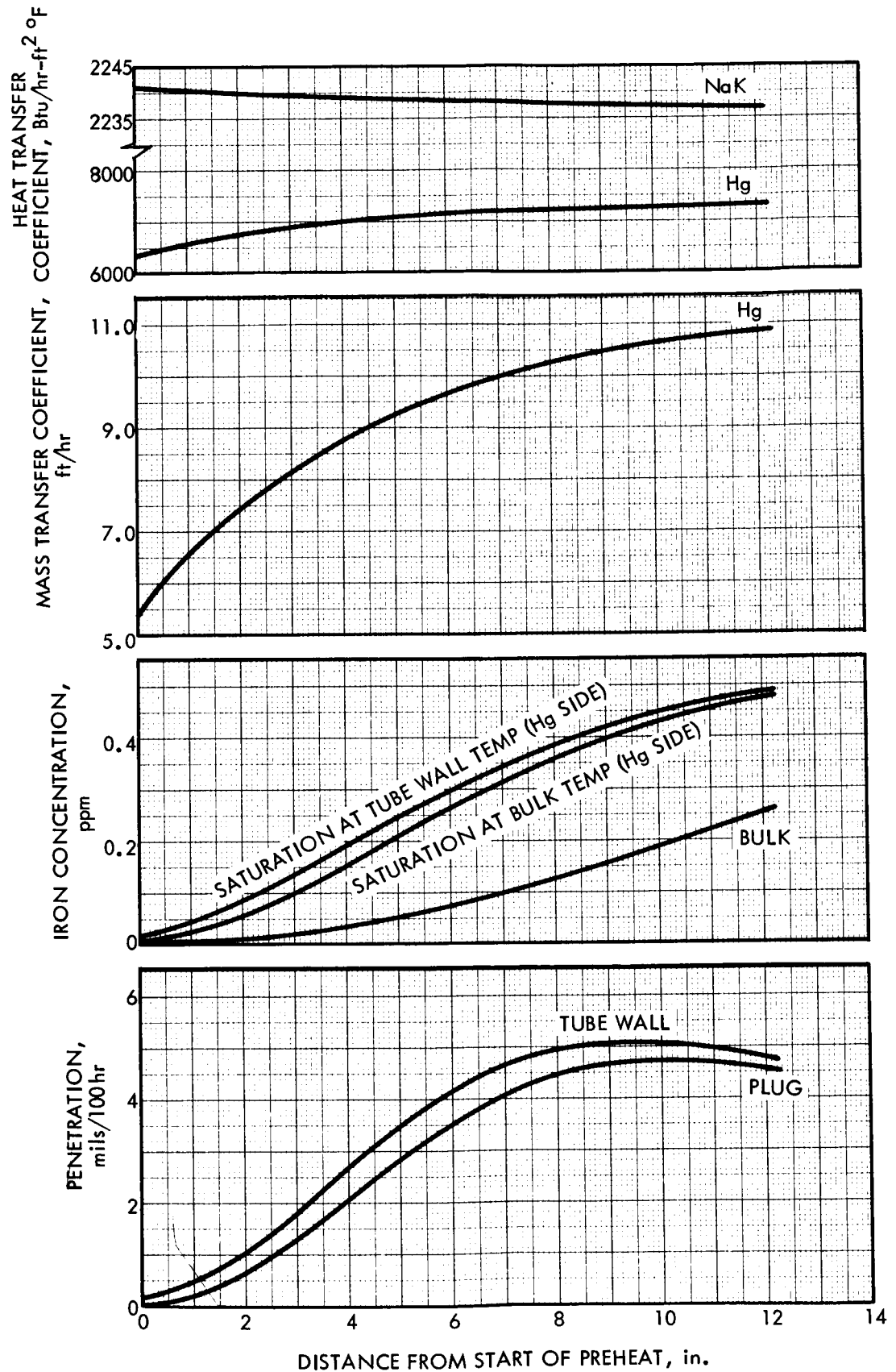
PHOTO MICROGRAPHS - INSIDE DIAMETER OF 9Cr-1Mo TUBING

CL 4 BOILER INLET SECTION

*Distance from Mercury Inlet

Figure 17

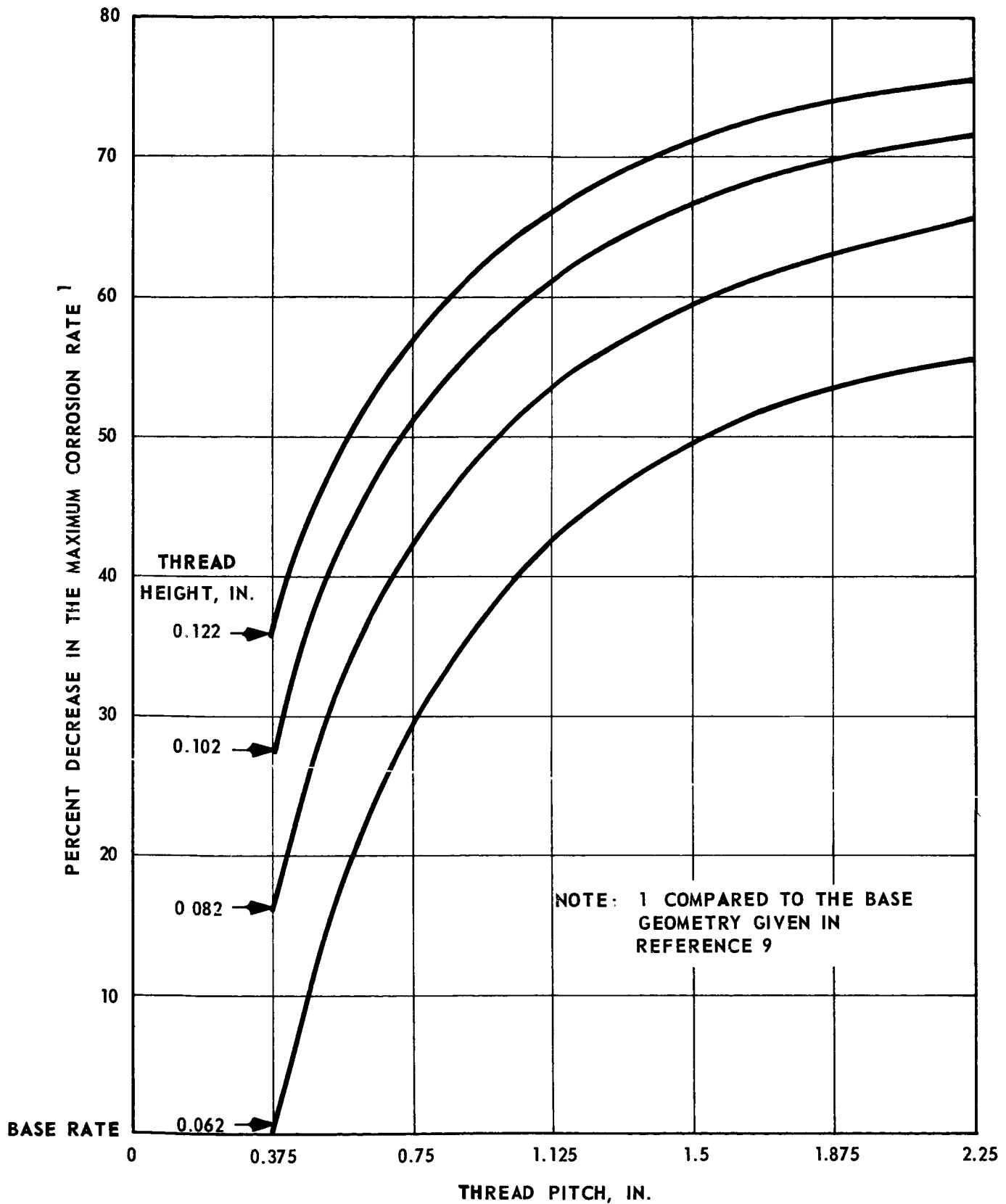
866-NF-1126



Axial Distribution of Variables Base Case

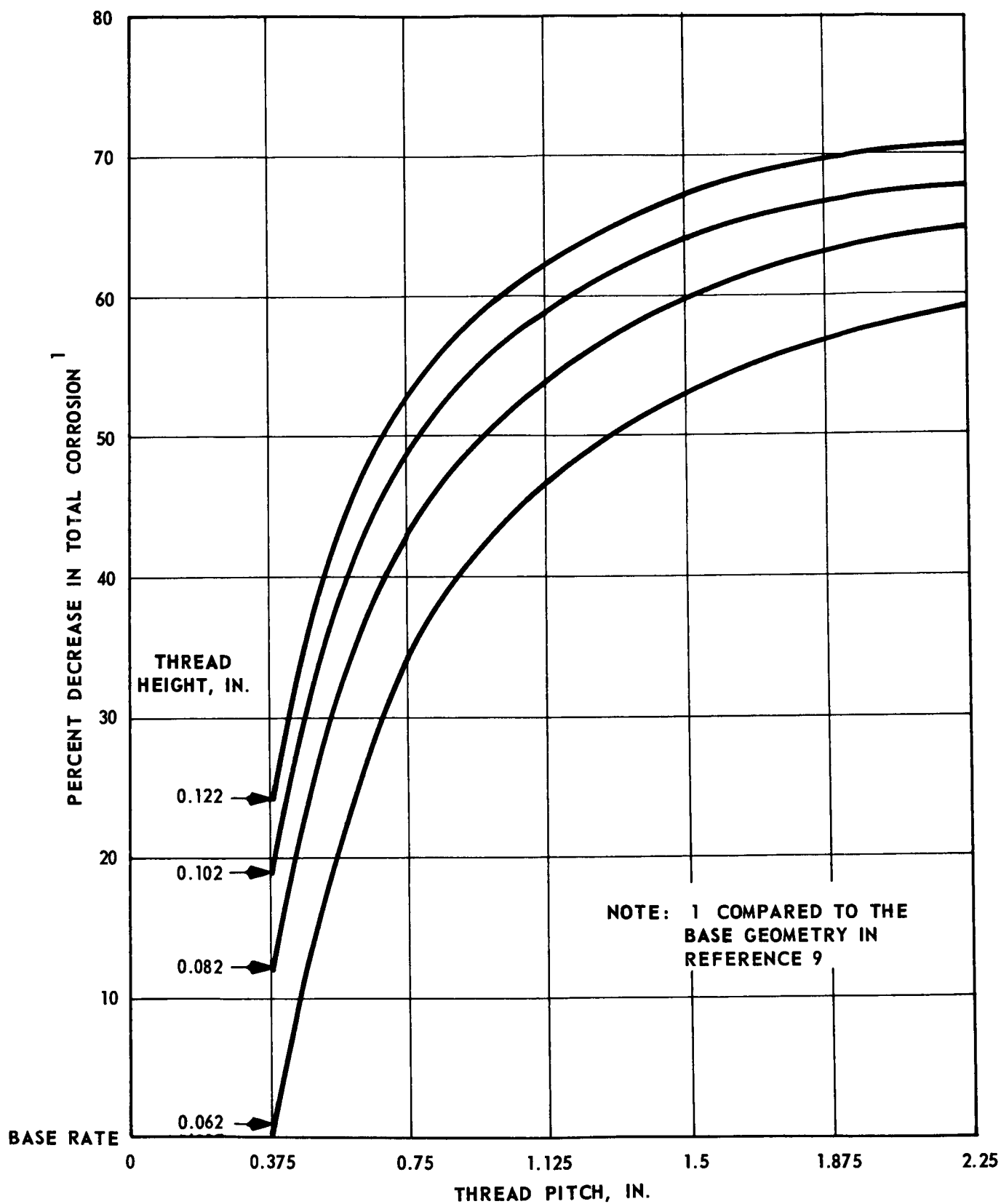
Figure 18

866-NF-1127/A



Effect of Thread Pitch and Height on the Maximum Corrosion Rate

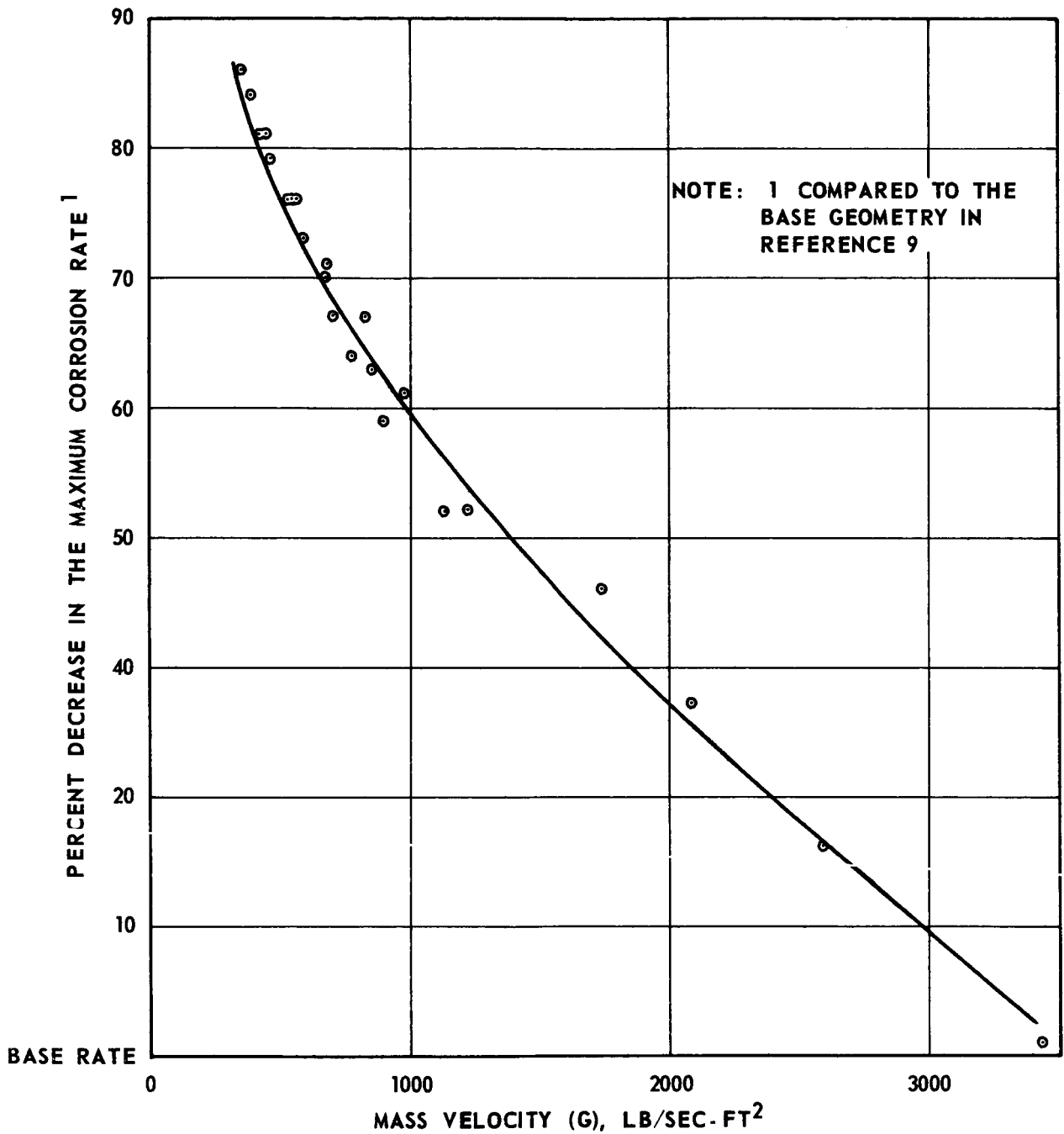
Figure 19



Effect of Thread Pitch and Height on the Total Corrosion

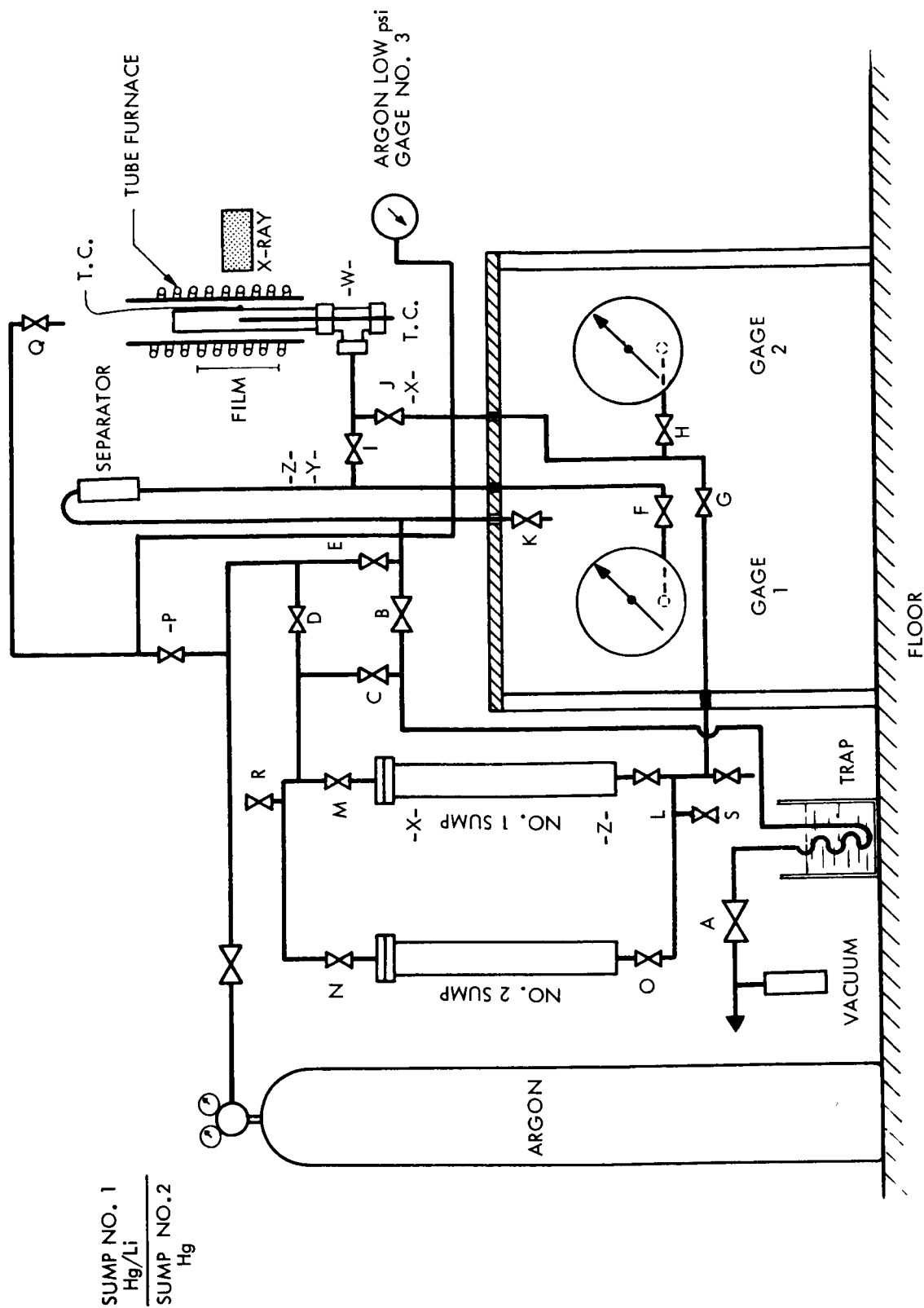
Figure 20

866-NF-1129/B



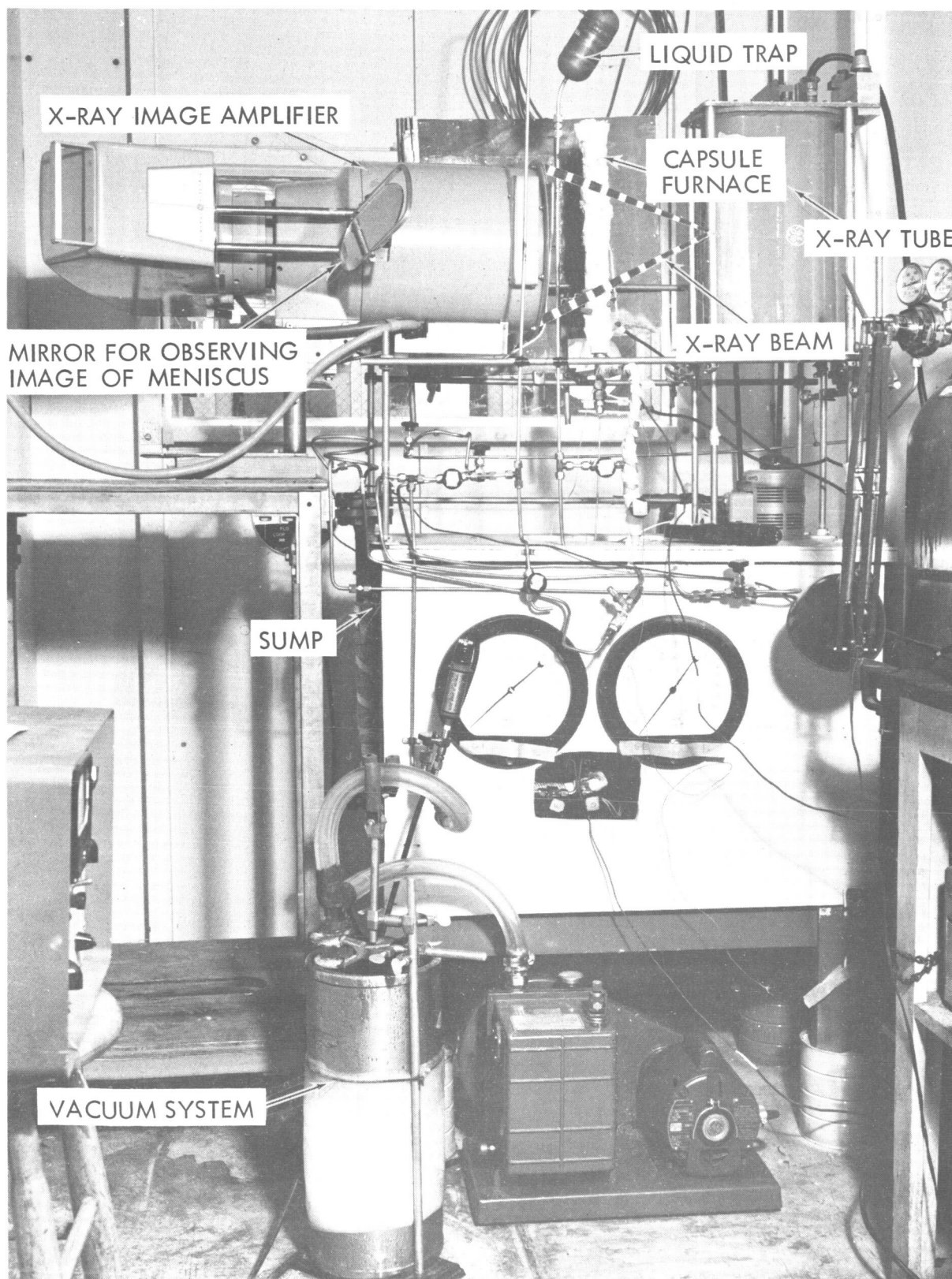
EFFECT OF MASS VELOCITY (G) ON THE MAXIMUM CORROSION RATE

866-NF-1130



SCHEMATIC OF MERCURY WETTING APPARATUS

Figure 22



Mercury Wetting Apparatus

Figure 23

866-NF-1131

866-NF-1132

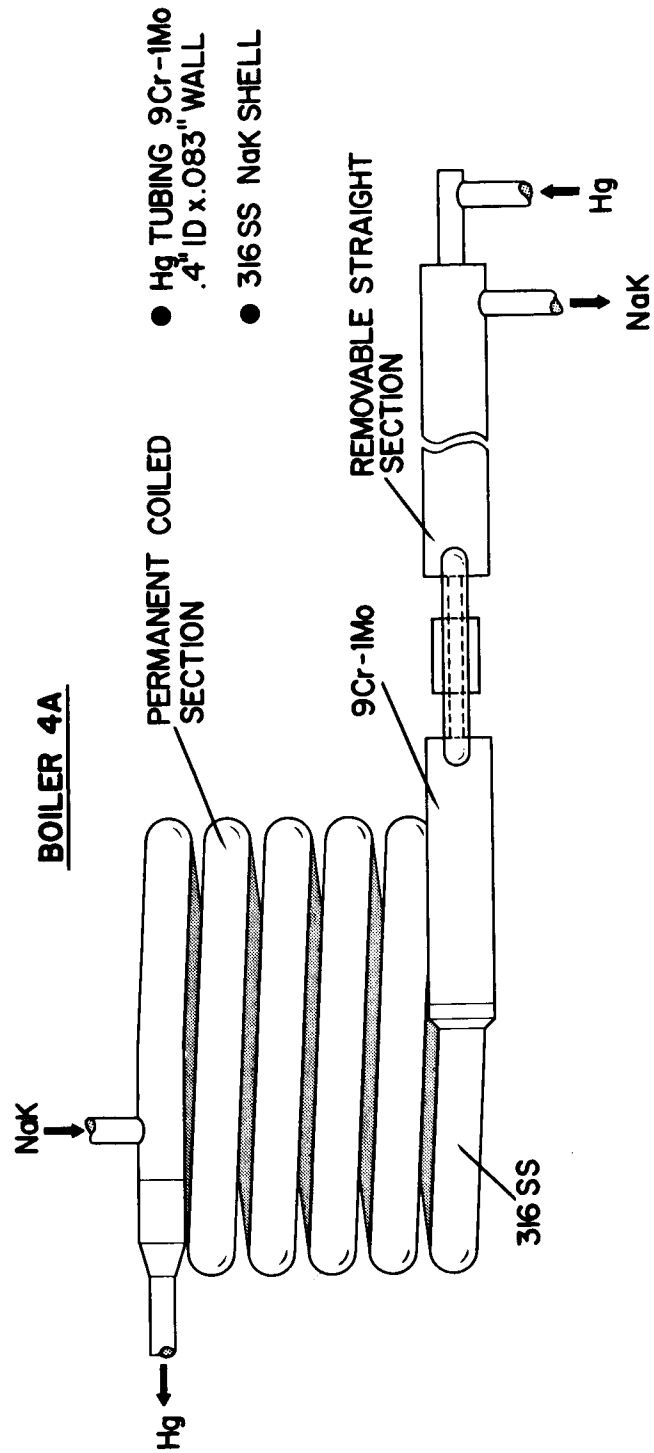


Figure 24

SCHEMATIC OF THE 4A BOILER

866-NF-1133

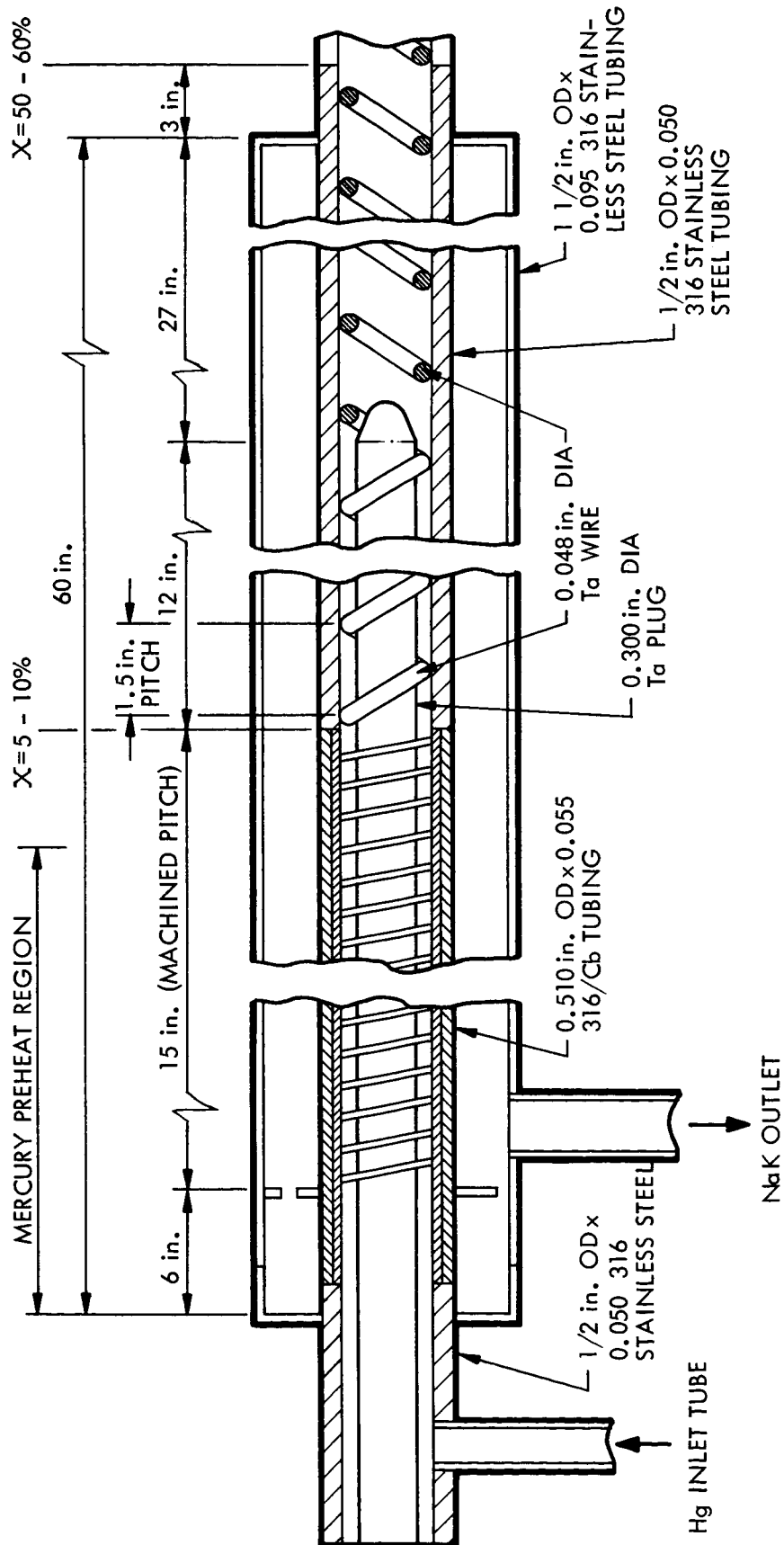
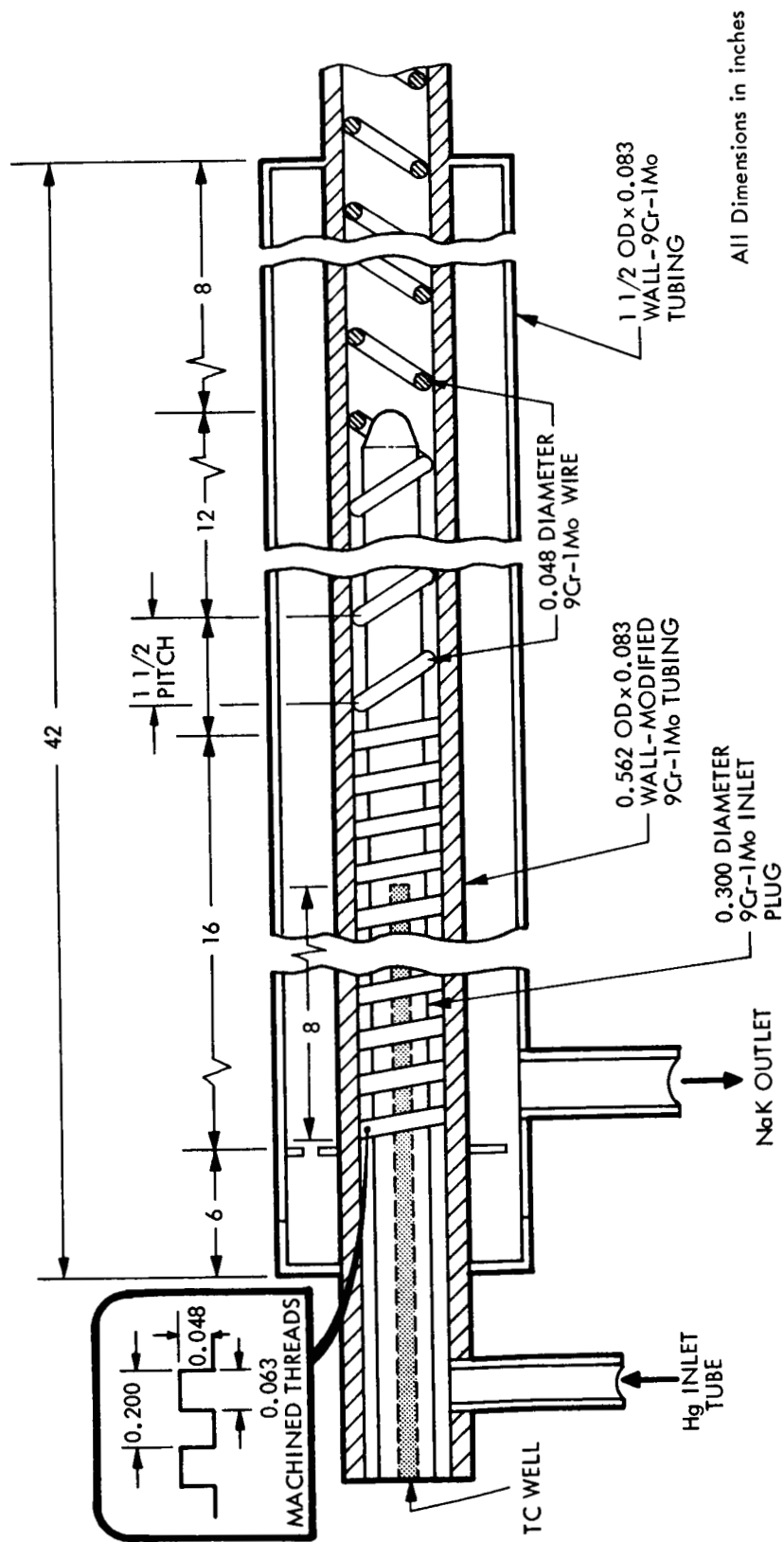


Figure 25

4A-2 TEST SECTION

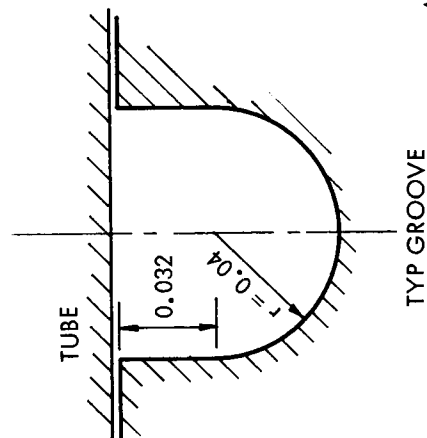
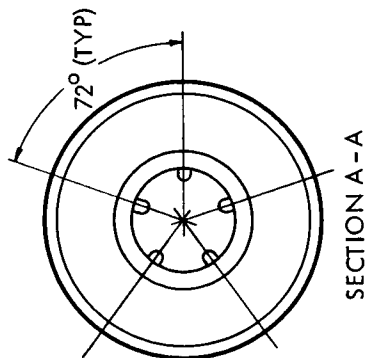
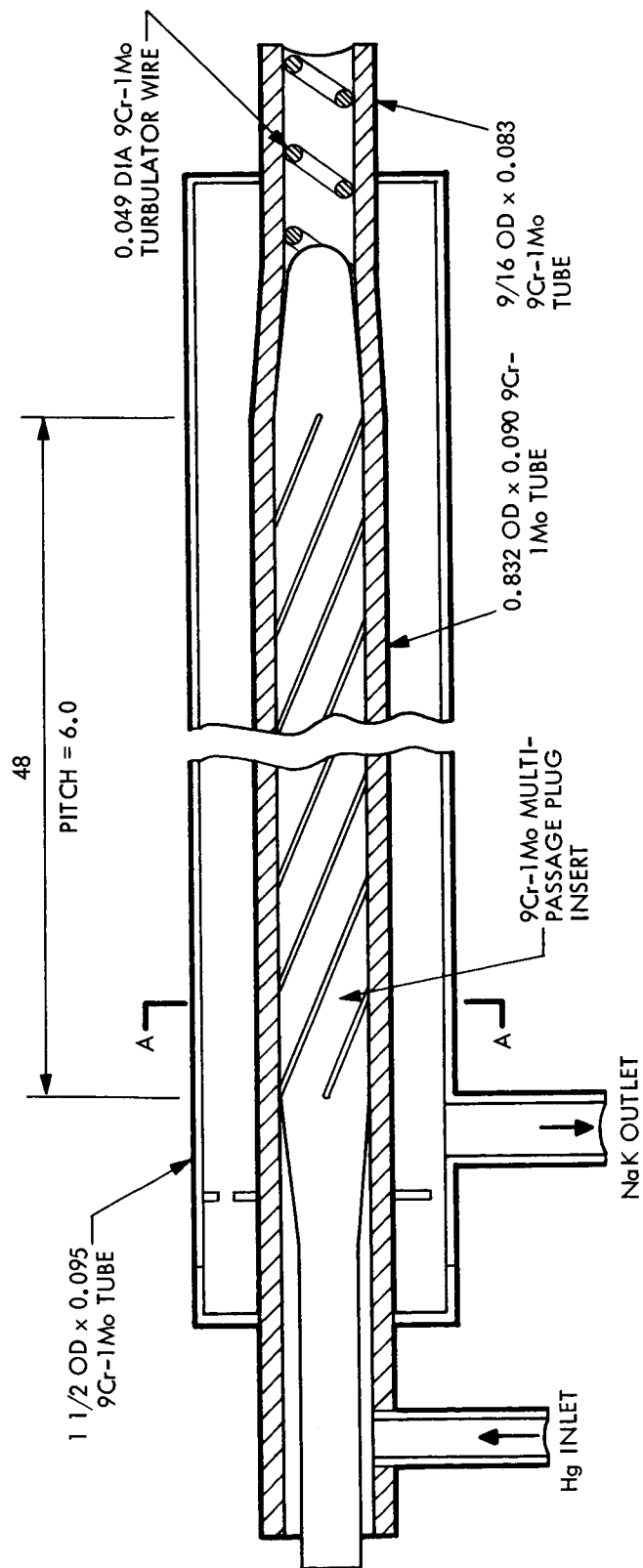
866-NF-1134



4A-3 TEST SECTION

Figure 26

866-NF-1135



All Dimensions in inches

4A-1 TEST SECTION

Figure 27

866-NF-1136

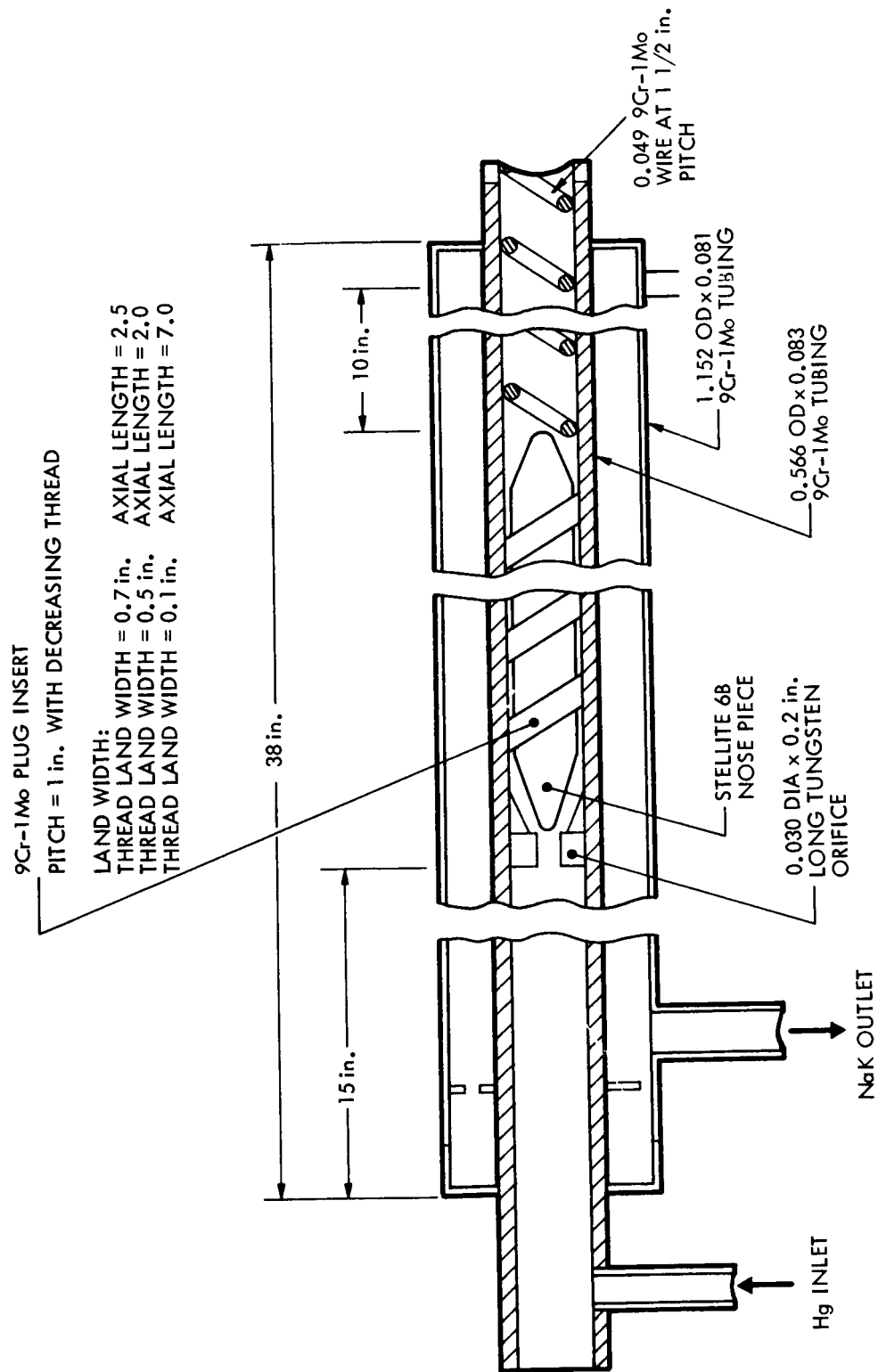
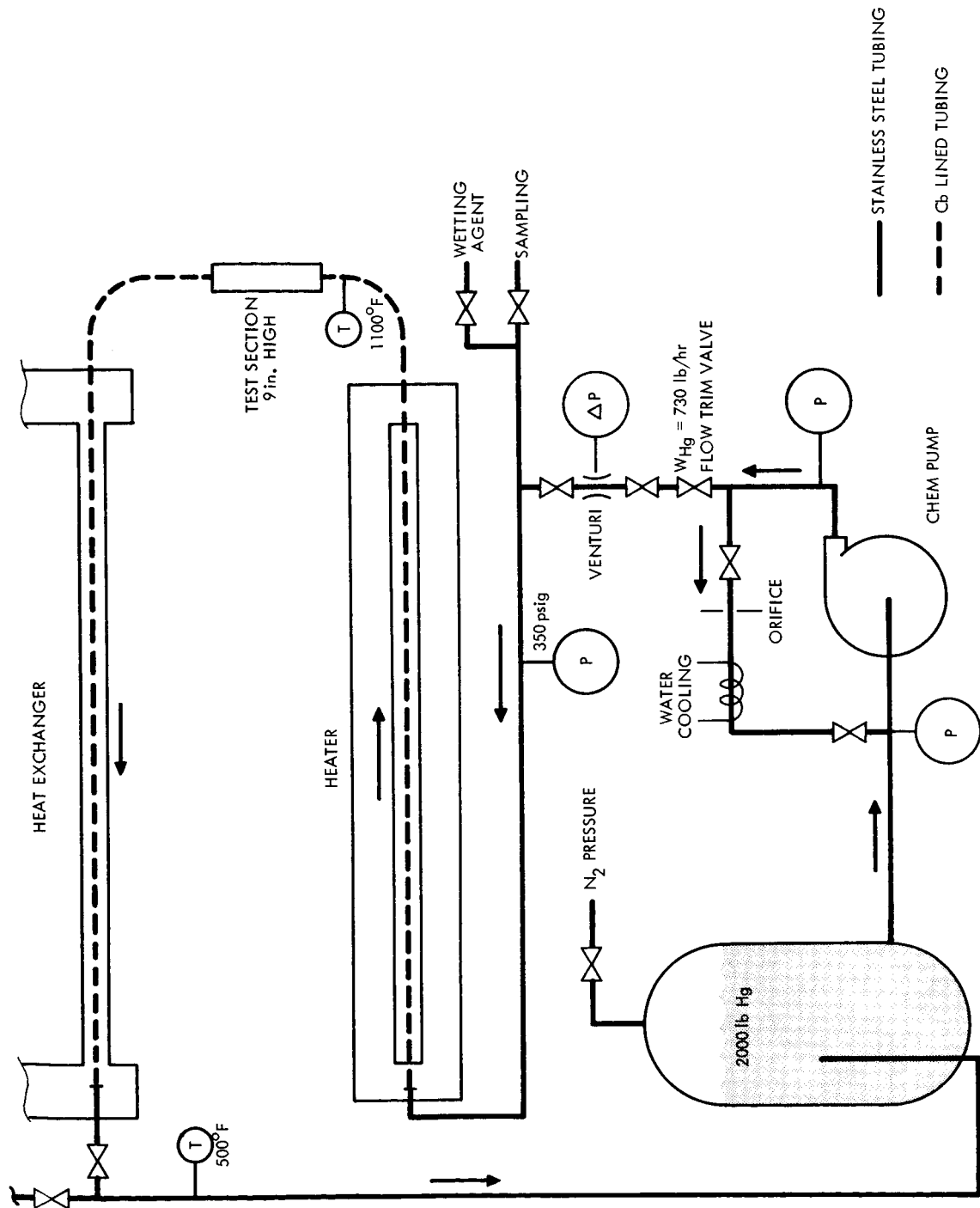


Figure 28

866-NF-1137



CML-1 P & I DIAGRAM

Figure 29

866-NF-1138

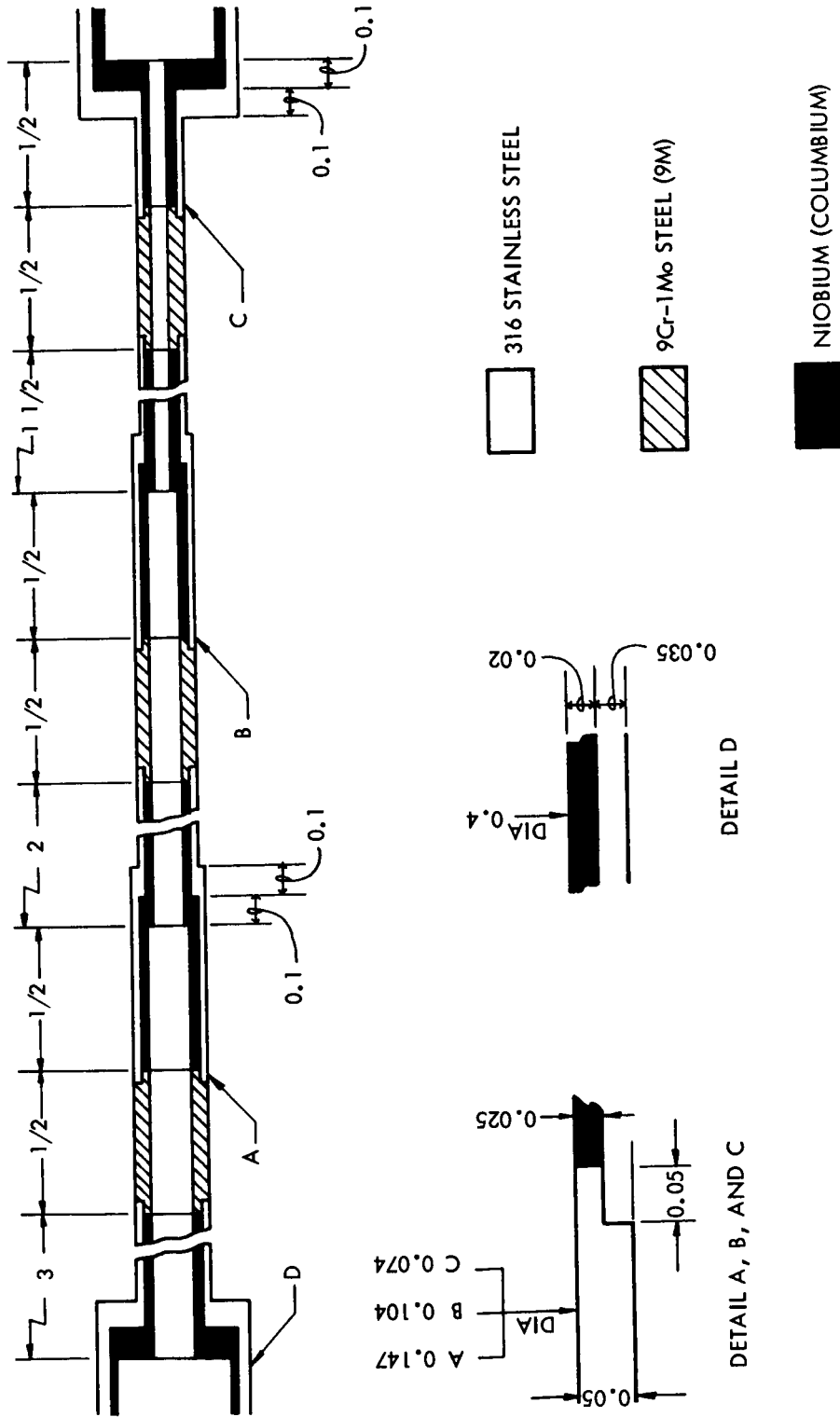


Figure 30

866-NF-1139

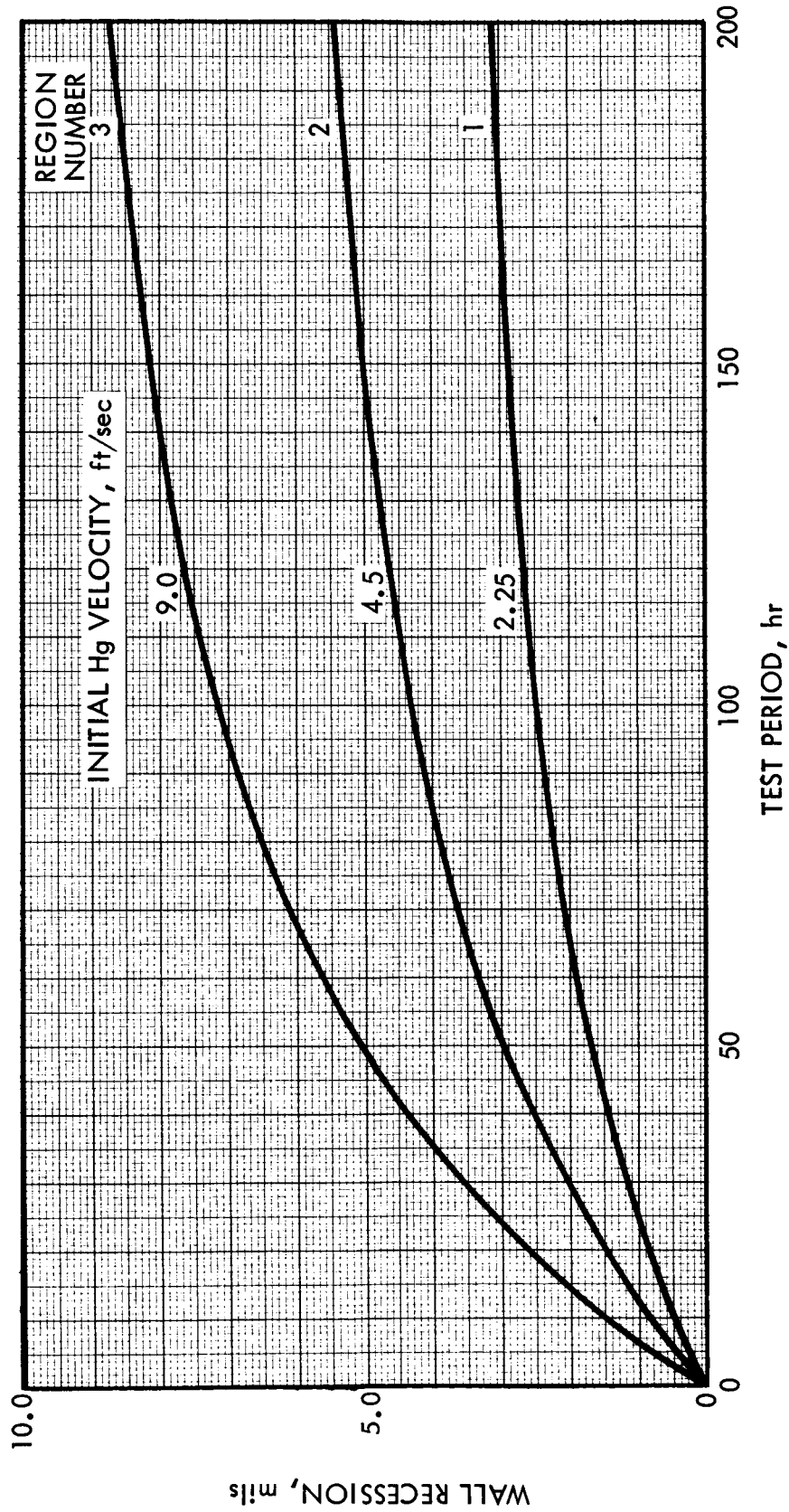


Figure 31

CML-1 TEST SECTION CORROSION PERFORMANCE (9M SAMPLES)